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DEVELOPMENT AND DEMONSTRATION OF MANUFACTURING PROCESSES FOR FABRICATING GRAPHITE/LARC-160 POLYIMIDE STRUCTURAL ELEMENTS

8TH QUARTERLY PROGRESS REPORT

MARCH 16, 1980 THROUGH JUNE 15, 1980

CONTRACT NAS1-15371

PART IV, PARAGRAPH C

PREPARED FOR
MATERIALS DIVISION
NASA LANGLEY RESEARCH CENTER
HAMPTON, VA.

(NASA-CR-159303-Pt-4) DEVELOPMENT AND DEMONSTRATION OF MANUFACTURING PROCESSES FOR FABRICATING GRAPHITE/LARC-160 POLYIMIDE STRUCTURAL ELEMENTS, PART 4, PARAGRAPH C Quarterly (Rockwell International Corp.,

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Space Transportation System
Development & Production Division
Space Systems Group

Rockwell International

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FOREWARD

This quarterly technical report was prepared by the Space Systems Group of Rockwell International, under contract NAS1-15371 for the Materials Application Branch, Materials Division, NASA Langley Research Center, Hampton, Virginia. Mr. Robert M. Baucom is the NASA Program Manager.

The following personnel have contributed to the work performed during this reporting period:

J. D. Leahy	Program Manager
R. K. Frost	Project Engineer
J. H. Bosler	Quality Control - NDI
R. J. Demonet	Physical Testing
P. J. Dynes	Materials Technology
C. L. Hamermesh	Materials Technology
D. W. Houston	Materials Technology
J. S. Jones	Process Development
R. L. Long	Specifications Development
A. A. Stenersen	Materials Technology

J. D. Leahy
Program Manager

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1.0 INTRODUCTION

Contract NAS1-15371 is the third NASA/LaRC program of Project CASTS, Composites for Advanced Space Transportation Systems. The first program utilized NR150-B2 polyimide resin and was conducted by General Dynamics, San Diego. PMR-15 polyimide resin was the subject of the second program conducted by Boeing Aerospace Company. The third program, NAS1-15371, utilizing LARC-160 polyimide resin, was awarded to the Space Systems Group of Rockwell International.

The three programs have as a common objective, the development and demonstration of technologies to implement structural application of graphite polyimide for 316C (600F) service environment. Technologies evolved from these programs will be transferred to Space Shuttle structural flight hardware according weight savings not attainable with conventional composite materials.

2.0 PROGRAM PLAN

The program is divided into two parts: process development and demonstration components. Each consists of several tasks. The program schedule is presented in Figure 1. The following briefly describes the objective of each task:

Part 1. Process Development

Task (a) - Develop a quality assurance program including specification for Celion/LARC-160 polyimide materials, quality control of materials and processes, including studies of the effects of monomer and/or polymer variables on the processibility of Celion/LARC-160 prepreg and on the mechanical properties of test specimens fabricated from the prepreg, and NDI of fabricated components.

Task (b) - Develop processes for fabricating laminates, hat and "I" stiffeners, honeycomb core panels, and chopped fiber moldings.

Task (c) - Fabricate specimens and conduct tests to qualify the processes for fabrication of demonstration components.

Part 2. Demonstration Components

Task (d) - Fabricate and NDI three (3) laminates 61x122-cm (24x48-in.) with 0, $\pm 45^{\circ}$ lay up symmetrical about the neutral axis. Laminate thickness will be 0.08 cm, 0.15cm, and 0.32cm (0.030 in., 0.060 in., and 0.125 in.).

Task (e) - Fabricate and NDI three (3) secondarily bonded hat-stiffened skin-stringer panels 23cm (9 in.) wide x 122cm (48 in.) long with 3 lengthwise stiffeners.

Task (f) - Fabricate six (6) honeycomb core panels 25.4x25.4-cm (10x10-in.) with 0.15cm (0.060 in.) thick face sheets with 0^{o}, 90^{o}, layup symmetrical about the neutral axis of the panel. The honeycomb core will be 2.54cm (1 in.) thick.

Task (g) - Fabricate six (6) chopped fiber moldings according to a specimen design mutually agreeable to Contractor and Contracting Officer's technical representative.

Task (h)- Fabricate a representative component of a Space Shuttle aft body flap that is mutually agreeable to the Contractor and Contracting Officer's technical representative.

3.0 PROGRAM PROGRESS

3.1 Task (a) - QUALITY ASSURANCE PROGRAM

3.1.1 Resin Variables Study Program

Three 4.5Kg (10 lb) batches of 30.4cm (12 inch) wide unidirectional tape have been received. These were produced under production conditions with separate batches of resin formulated for each 4.5 Kg (10 lbs.) of prepreg and will be used to demonstrate formulation repeatability and material usability over a six month period at -18C(0F) storage and after ambient out-time exposure of seven days.

The resin batches were formulated within limits established from evaluating thirteen batches of prepreg, as noted in the 7tn Quarterly Reports, which varied in stoichiometry and processing. Formulation and processing limits for the three batches were as follows:

AP-22 $\pm$ 2.5% by weight

NA $\pm$ 2.5% by weight

BTDA $\pm$ 2.5 by weight

Reflux time - not to exceed 90 minutes at 82 $\pm$ 3C (180 $\pm$ 5F)

Cook time - not to exceed 115 minutes at 82 $\pm$ 3C (180 $\pm$ 5F)

Prepreg material physical properties specified were:

Resin solids: 37 ± 3%

Volatiles: 12 ± 3%

Fiber Areal Weight: 134 ± 3 $grams/m^2$

Samples of the neat resin and intermediate ester were provided with each batch of prepreg. These will be used for HPLC analysis to determine repeatability and establish a standard for comparison of resin extracted from the prepreg during the storage period. The proposed test program was presented in the 7th Quarterly Report.

3.2 Task (b) - PROCESS DEVELOPMENT

3.2.1 Celion/LARC-160 Process Improvement

Process improvement studies, initiated under NASA/LaRC NAS1-[illegible]83 (Task 14), were further developed under this contract to simplify the imidizing and autoclave cure cycles. Target improvements were as follows:

(1) Reduce the number of steps in the imidizing cycle.

(2) Increase the prepreg preform imidizing temperature and/or time at temperature, thereby increasing the LARC-160 resin viscosity when hot melt occurs during the cure cycle. This would allow application of 1378 KN/m^2 (200 psi) pressure at the initiation of cure at room temperature and, in turn, eliminate the chance of error of pressure application in the temperature range of 274C to 287C (525F to 550F) with the existing cure cycle. Allowing the resin to seek its normal flow point while under constant pressure, also eliminates problems related to temperature non-uniformity due to varying part thickness or tooling mass.

(3) Eliminate the intermediate 163C (325F) steps in the autoclave cure cycle by raising the temperature from room temperature to final cure temperature within the established heat rise rate band.

3.2.2 Process Improvement Procedures and Results

One laminate panel 30.5x203-cm (12x80-inches) was laid up 26 plies thick using U.S. Polymeric batch 2W4612 nominal 67 ± 3 $grams/m^2$ fiber weight prepreg. Physical properties are given in Table 1. Celion fibers employed standard Celanese epoxy resin sizing. The stacked laminate was vacuum bag debulked per the detailed procedures described in the 7th Quarterly Report. Bleeder materials, comprised of one ply 120 fiberglass top surface and two plies bottom surface, were installed as an integral part of the preform during the debulking operation. The debulked and consolidated preform was cut into individual panels, 15.2x15.2-cm (6x6-inches), for the imidizing study defined in the process development matrix, Table 2.

The debulked laminates were imidized in groups of five per Table 2 and the cycle per Figure 2 on standard perforated tooling defined in the 7th Quarterly Report. After imidizing, each laminate was carefully sectioned thru the center parallel to $(0)_{26}$ direction to provide a 1.27 cm (0.50 inch) wide strip for materials properties testing. Resin content and volatile samples were extracted from sections of each strip and tests performed. Results of these tests are presented in Table 1. Significant results showed residual volatiles to be < 2% on all staged panels indicating complete imidization occurs at 163C (325F) within 60 minutes. Panels exposed for longer times and higher temperatures showed no significant decrease in volatile content from those exposed for lesser times and lower temperatures.

The sectioned panels were carefully reassembled at the center line with bleeders in place and prepared for curing on standard flat laminate tooling described in the 7th Quarterly Report. The modified panel size was 15.2x13.9-cm (6x5.5-inches).

Twenty-five imidized panels were autoclave cured under the same bag per the cure cycle shown in Figure 2. Autoclave pressure of 1378 KN/m^2 (200 psi) was applied at the start of cure at room temperature. Actual heat rise rate for the cure cycle averaged 1.3 (2.3F) minute. Panels were submitted for NDI C-scan testing and specimens removed for physical properties tests.

Panels were postcured 4 hours at 316C (600F), in an air circulating oven using standard procedures described in the 7th Quarterly Report. During this process the oven controls malfunctioned and specimens were destroyed when the temperature reached approximately 482C(900F).

The following observations were made on panels cured using the improved cycle.

(1) High resin flow was noted on all panels imidized at 162 and 177C (325 and 350F) as indicated by saturation of surrounding fiberglass breather layers. Panels imidized at 191 and 199C (375 and 390C) showed excellent compaction characteristics and good resin beading at laminate edges. The panels imidized at 218 (425F) had minimal evidence of resin flow. The panels imidized for 30 and 60 minutes had overall excellent cosmetic appearance while those imidized for longer periods at 218C (425F) showed surface roughness discrepancies indicating inadequate resin flow.

(2) NDI C-scan testing results showed all panels imidized at 162C (325F) for time periods of 60, 90, 120, 150 and 180 minutes had void area discrepancies ranging between 40 and 80%. Void area increased with lesser time at imidizing temperature. Panels imidized at 177C (350F) showed ultra sound penetration improvement starting at 150 minutes with approximately 7% void area showing. Panels imidized at 191C (375F) starting at 60 minutes thru 150 minutes showed 100% ultra sound transmission except

for some cellulose acetate fiber splice voids. All panels imidized at 199 and 218C (390 and 425F) had 100% ultra sound transmission. As previously noted, panels imidizing at 218C (425F) for time periods exceeding 60 minutes showed surface roughness irregularities due to resin flow reduction.

NDI C-scan recordings of each panel evaluated are presented in Figures 3 thru 27.

(3) Physical properties testing results verified imidizing time/temperature relation as observed in panel cosmetic appearance and NDI C-scan tests. All panels imidized at 163 and 177 C (325 and 350 F) had high fiber and void volumes. Panels imidized at 199 and 202C (395 and 390F) for time periods between 30 and 150 minutes achieved target fiber volume of 60 $\pm$ 2% void volume $<$ 2%. Those panels imidized at 218C (425F) for 30 and 60 minutes also achieved target requirements. Panels imidized at 218C (425F) for longer time periods had low fiber volumes; void volumes were $<$ 2%. Detailed physical properties are presented in Table I.

3.2.2.7 Results of this phase of the process improvement study indicate the best imidizing time/temperature bands to be in the ranges of 191C (375 F) for 60 to 150 minutes, 199C (390F) for 30 to 150 minutes and 218 C (425 F) for 30 to 60 minutes. It is planned to fabricate panels by imidizing at the extreme ends of each of the above time/temperature cycles. Panels 30.5x30.5x0.20-cm (12x12x0.08-inch) will have a (0, $\pm$ 45, 90)s fiber orientation and panels 15.2x15.2x0.165-cm (6.0x6.0x0.065-inch), a $(0)_{26}$ orientation. All panels will be cured under the same vacuum bag using the cure cycle shown in Figure 2. Criteria for evaluation will be based on:

(1) Maximum C-scan penetration at "A" sensitivity

(2) Fiber volume control to a target 60 $\pm$ 2%

(3) Measured voild volume: $<$ 1%.

(4) $(0)_{26}$ flexure and short beam shear properties equivalent to laminates fabricated using the standard process.

Based on these tests, a specific imidizing cycle will be selected. The selected process will be verified by fabricating "hat" stringers and "pi" joint elements.

3.3 TASK (c)-FABRICATION AND TEST

3.3.1 Standard Celion/LARC-160 Cure Process Verification

The standard procedures for layup, debulking, imidizing, and autoclave curing Celion/LARC-160 flat laminates described in the 7th Quarterly Report were verified during this reporting period.

Materials were supplied by Hexcel, U.S. Polymeric & Fiberite: eight batches of prepreg from the resin stoichiometry variable and processing program, Task (a) and three batches of standard prepreg materials with nominal ply thicknesses of 0.0635, 0.127, and 0.145mm (0.0025, 0.0005, and 0.0057 inch) per ply. Detailed prepreg physical properties and bleeder arrangement details are presented in Table 3. Eleven laminates, one for each material batch, measuring 30.5x30.5-cm (12x12-inches), and nominally .23-cm (.09 inches) thick were prepared in a (0, ± 45, 90)s orientation using the standard processing procedures. All panels were imidized at the same time under one bag and autoclave cured, again under the same bag.

Results of the process verification tests demonstrated:

(1) 100% C-scan "A" sensitivity ultra sound transmission through all laminates. C-scan test results are shown in Figures 28 thru 38.

(2) Conformance of 60 ± 2% fiber volume target with prepreg materials meeting resin content specification requirements. Laminate physical properties are given in Table 3.

(3) Measured void volumes <1%.

(4) A large latitude exists in LARC-160 resin stoichiometry and processing conditions where laminates can be successfully produced.

3.3.2 Honeycomb Sandwich Panel Elements

The last four sandwich elements required on the program were fabricated in accordance with processes described in the 5th Quarterly Report and prepared for test as described in the 7th Quarterly Report. NDI C-scan "A" sensitivity tests performed on $(0_2 \pm 45, 0)_t$ 5 ply, 0.145 mm/ply (5.7 mil/ply) skins verified high quality and are shown in Figures 39 and 40.

One panel, in the post cured (4 hours @ 316C (600F) condition, was tested in compression at -168 (-270F). The three remaining panels were aged for 125 hours at 316C (600F) before compression tested at -168 (-270F), RT and 316C (600F). All panels tested exceeded the room temperature design ultimate requirement of 120 KN (27,000 lbs) or 52.5 KN/(3000 lbs/inch). Failure modes were by skin compression in both face sheets usually near a base doubler. Structural elements are shown after test in Figures 41 thru 44. Axial load/strain curves were very linear thru failure and are presented in Figures 45 thru 48. Detailed compression test data are presented in table 4.

3.4 TASK (e) - SKIN/STRINGER PANEL FABRICATION

Three secondarily bonded hat-stiffened skin-stringer panels were fabricated per processes described in previous Quarterly Reports and delivered to NASA-LaRC during this reporting period. Panel design and fabrication processes were qualified in -132C, RT and 316C compression test of 23 x 30.5-cm (9.0 x 12.0 inches), subelements as described in the 2nd and 7th Quarterly

Reports.

The panels delivered measured 26x122-cm (10.2x48-inches) and incorporated three lengthwise stiffeners. Photographs showing the skin-stringer panels are presented in Figures 49 and 50. NDI C-scan recordings of the FM 34B-18 adhesive bonded stringer to skin bond areas are presented in Figures 51 and 52.

3.5 TASK (g) - CHOPPED FIBER MOLDING FABRICATION

Matched metal mold for fabricating individual flexure and tensile specimens have been obtained. Process development will start during the time period of the 17th Monthly Report.

3.6 TASK (h) - TECHNOLOGY DEMONSTRATOR SEGMENT

Materials purchased under this contract for fabricating sections of the graphite polyimide technology demonstrator segments (TDS) include 1) 27.9m^2 (300 ft^2) of .439 Kg/m^2 (.09 lb/ft^2) FM34B-18 polyimide adhesive from American Cyanimid and (2) 19 liters (5 gallons) of BR34B-18 primer, also from American Cyanimid. The latter material will be used not only as the adhesive primer but also as the basic ingredient for formulating potting material for reinforcing core in local high load areas such as around mechanical fasteners. All the above materials are being stored at -18C (0°F) and will be used with TDS fabrication status during the next quarter.

DEVELOPMENT OF CELION/LARC-160 STRUCTURAL ELEMENTS - NAS1-15371
PROGRAM SCHEDULE
(SCHEDULE BEYOND JUNE IN VERBAL NEGOTIATION FOR EXTENSION)

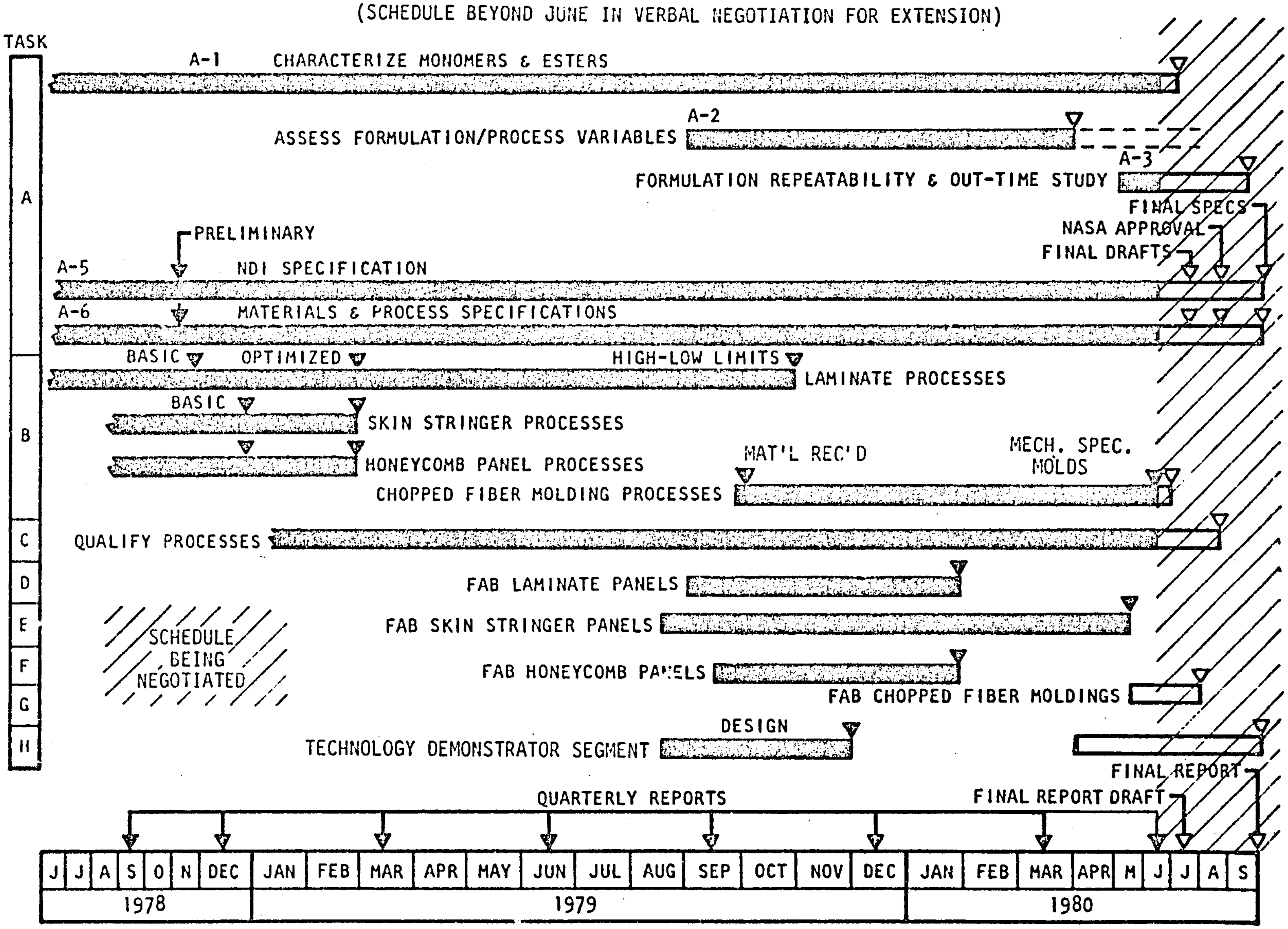

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Figure 1

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Rockwell International

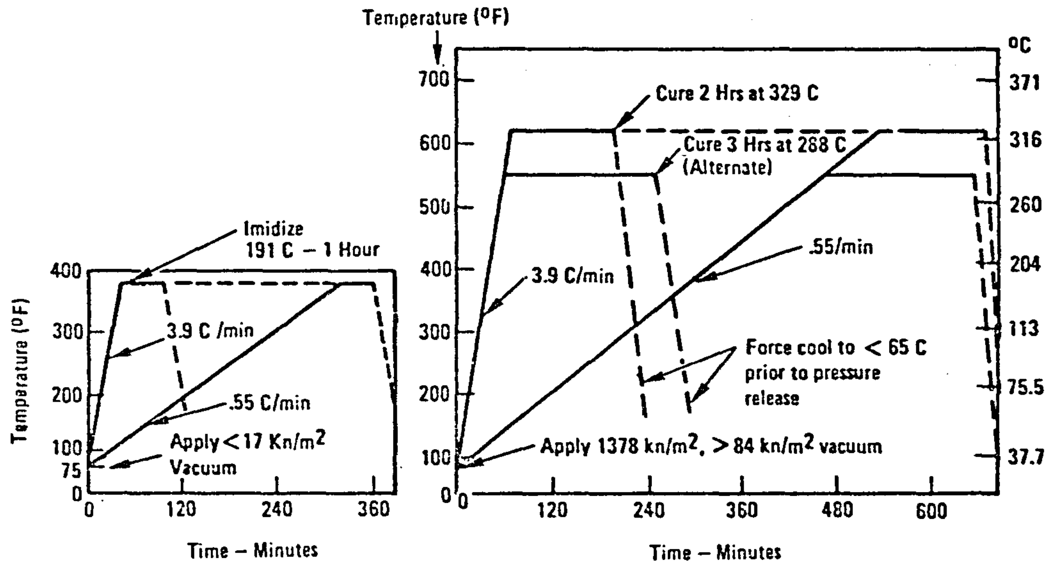

Figure 2. Improved Imidizing Cycle · Improved Cure Cycle

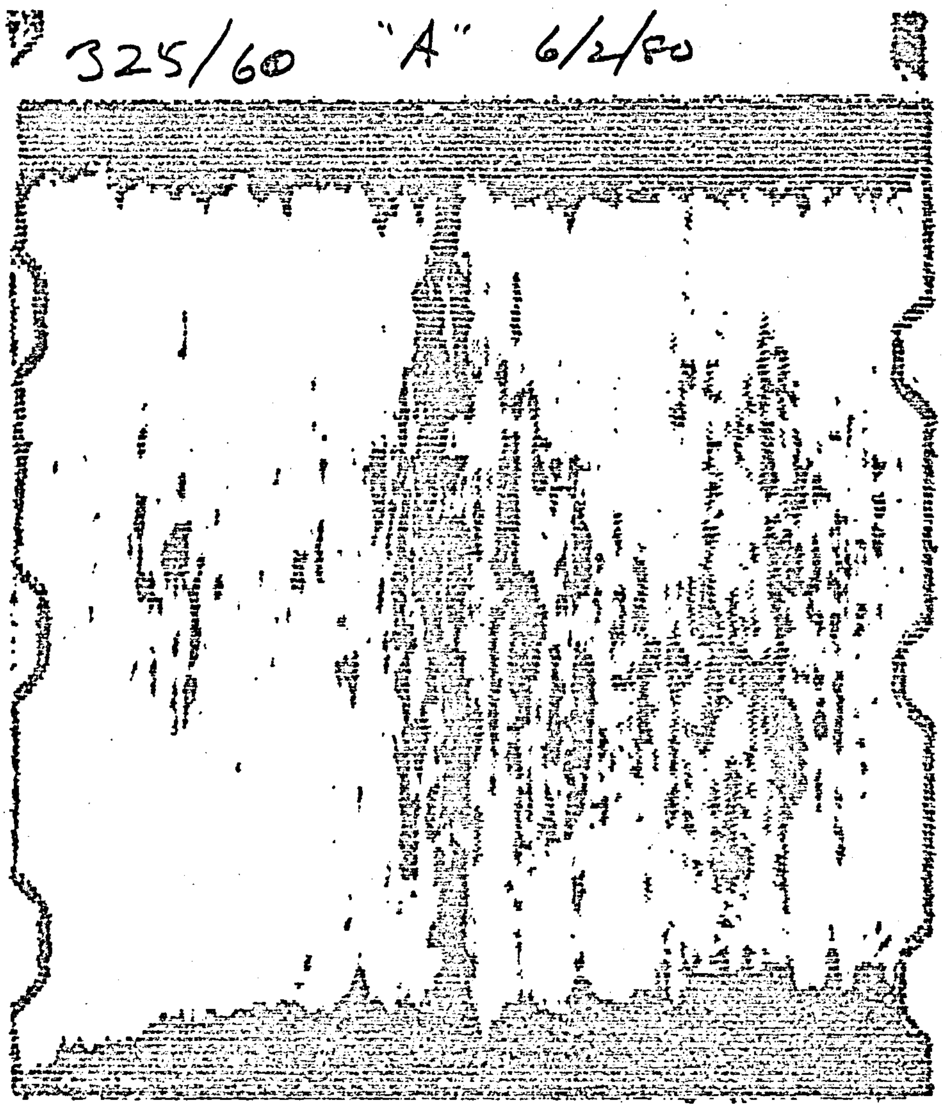

Figure 3. C-Scan of Laminate Imidized at 163 C, 60 Minutes

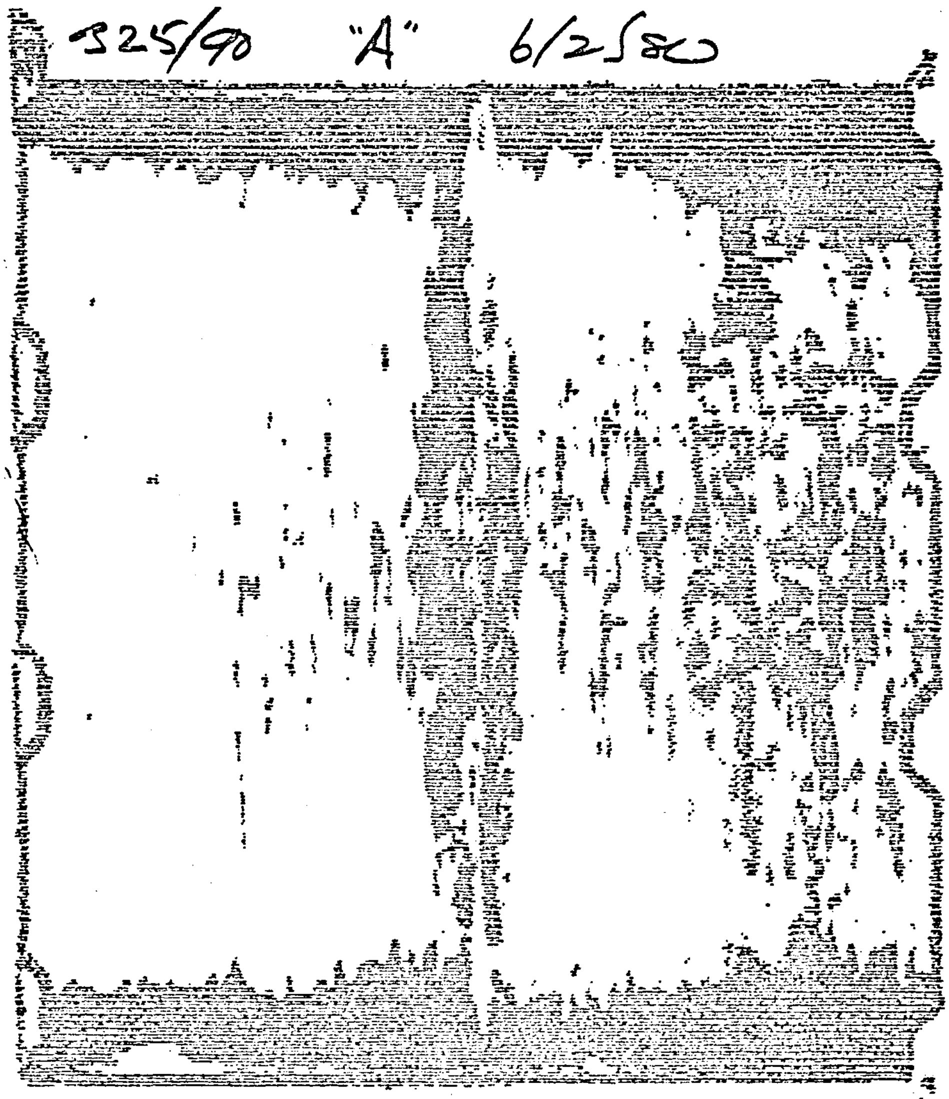

Figure 4. C-Scan of Laminate Imidized at 163 C, 90 Minutes

325/120 "A" 6/2/80

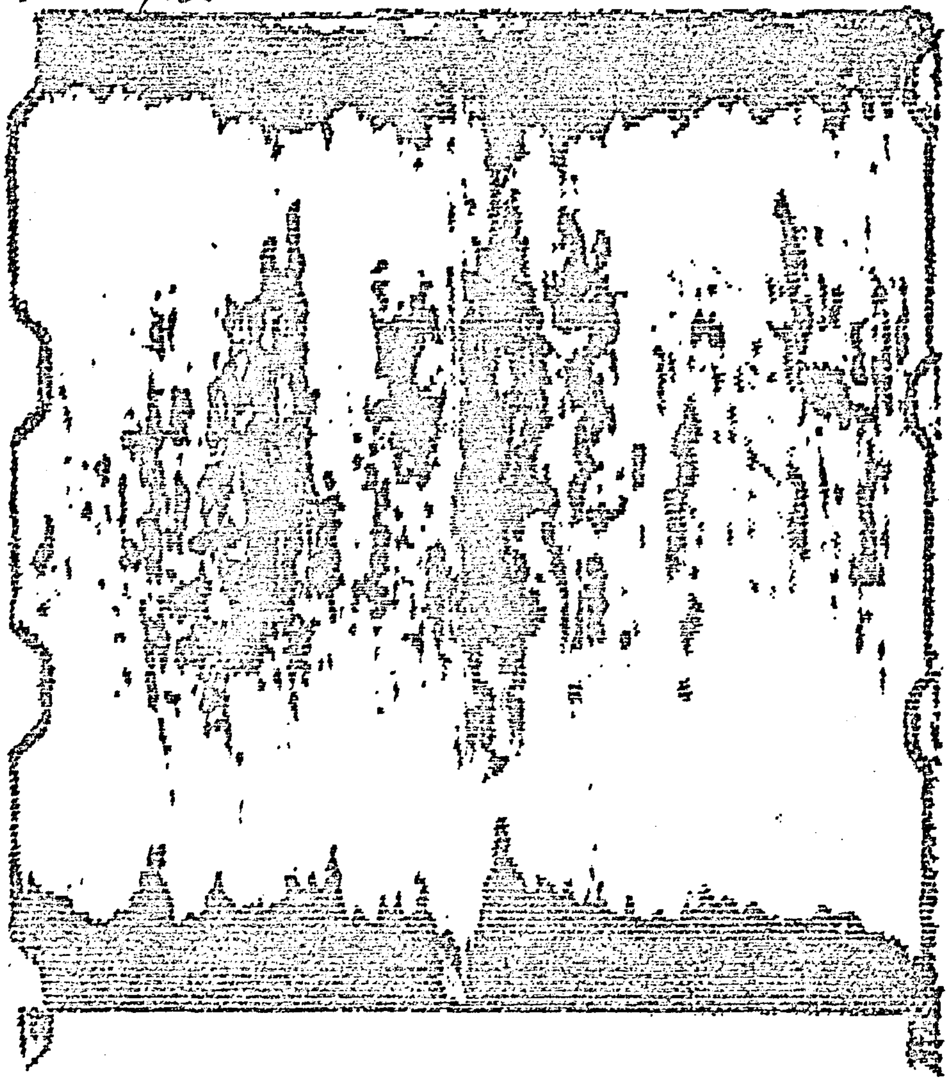

Figure 5. C-Scan of Laminate Imidized at 163 C, 120 Minutes

325/150 "A" 6/2/80

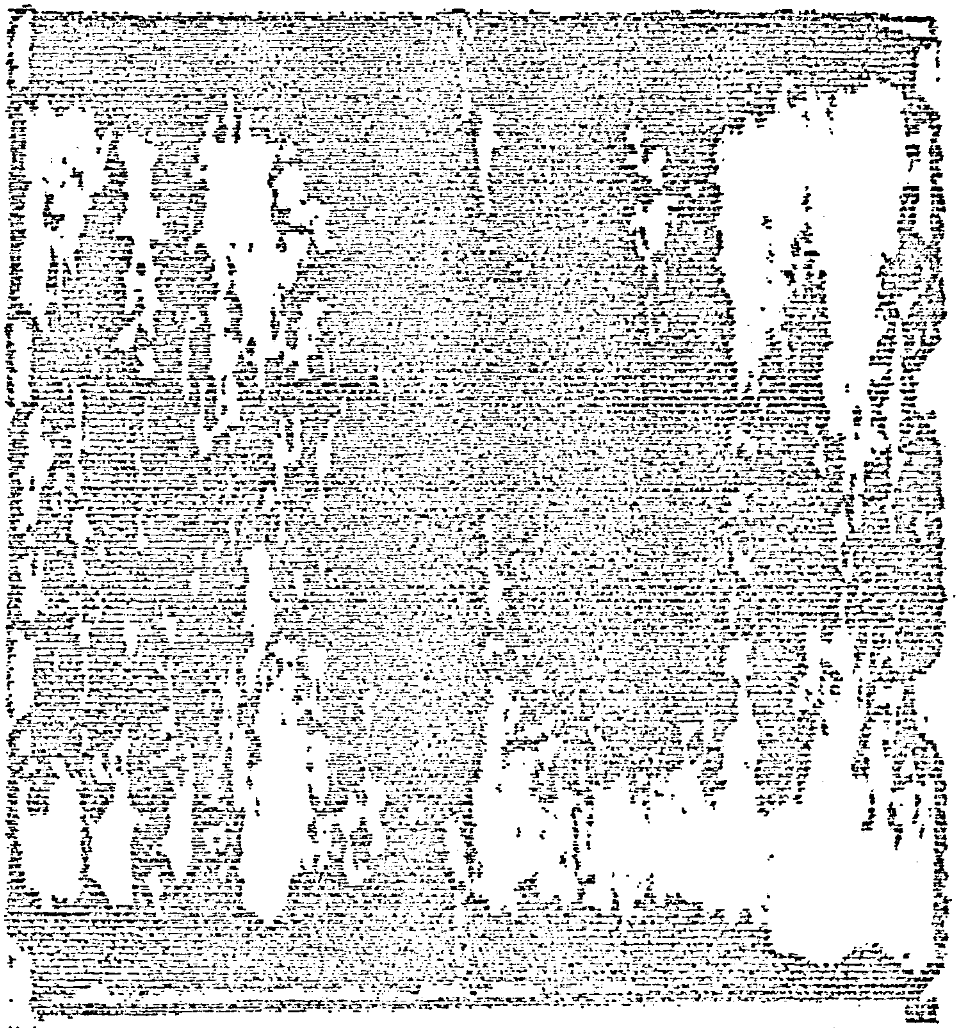

Figure 6. C-Scan of Laminate Imidized at 163 C, 150 Minutes

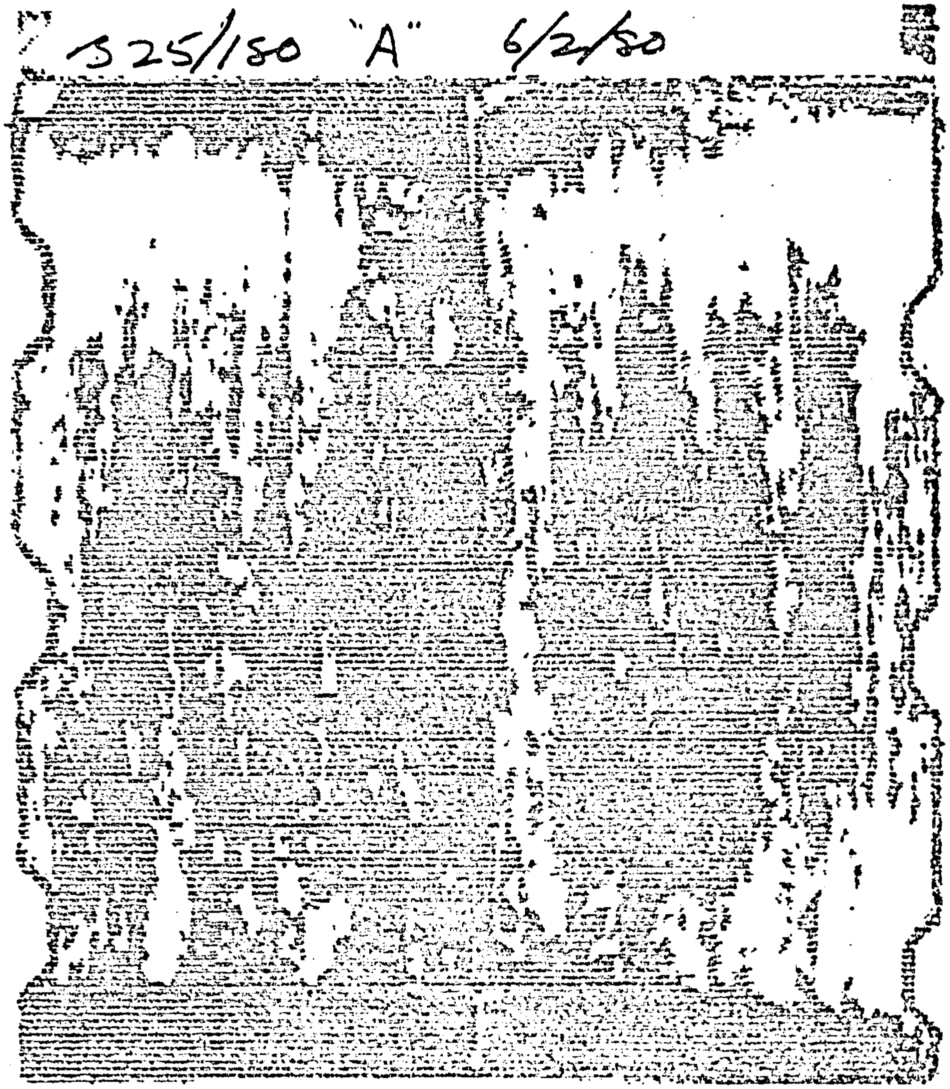

Figure 7. C-Scan of Laminate Imidized at 163 C, 180 Minutes

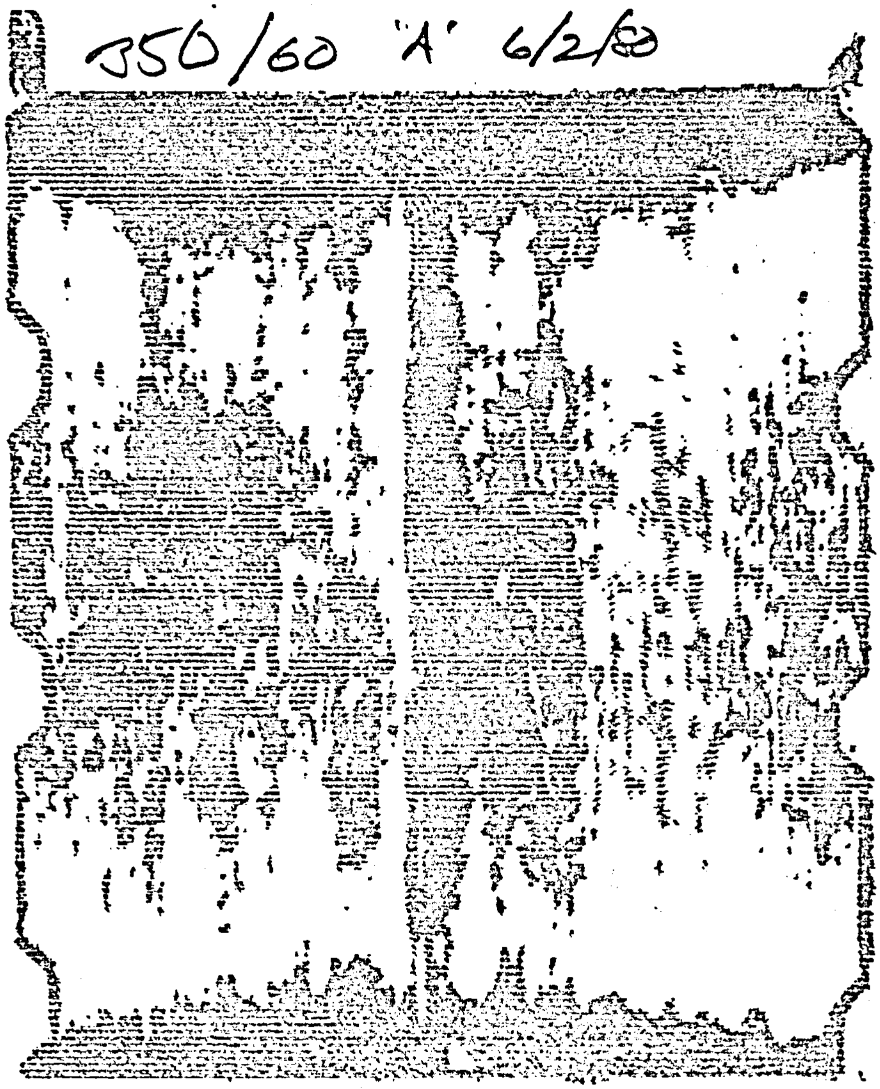

Figure 8. C-Scan of Laminate Imidized at 177 C, 60 Minutes

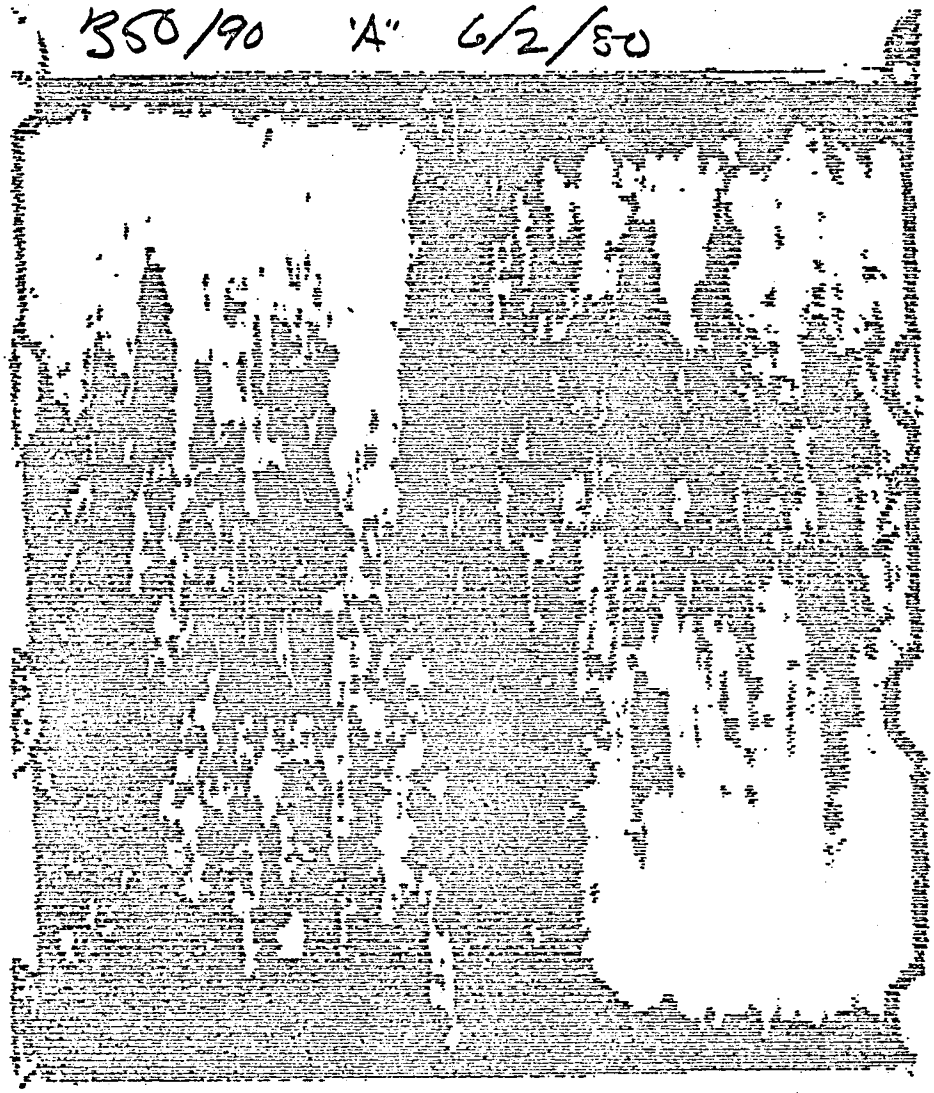

Figure 9. C-Scan of Laminate Imidized at 177 C, 90 Minutes

350/120 "A" 6/2/80

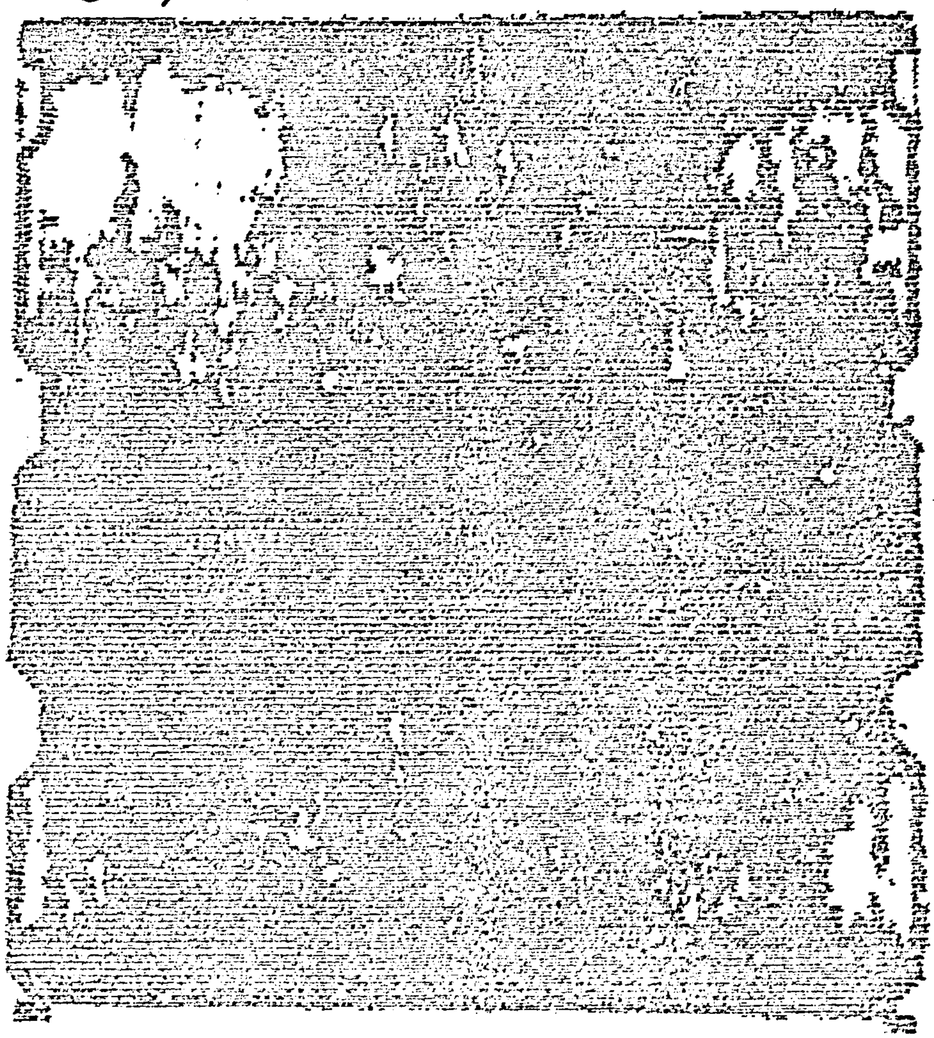

Figure 10. C-Scan of Laminate Imidized at 177 C, 120 Minutes

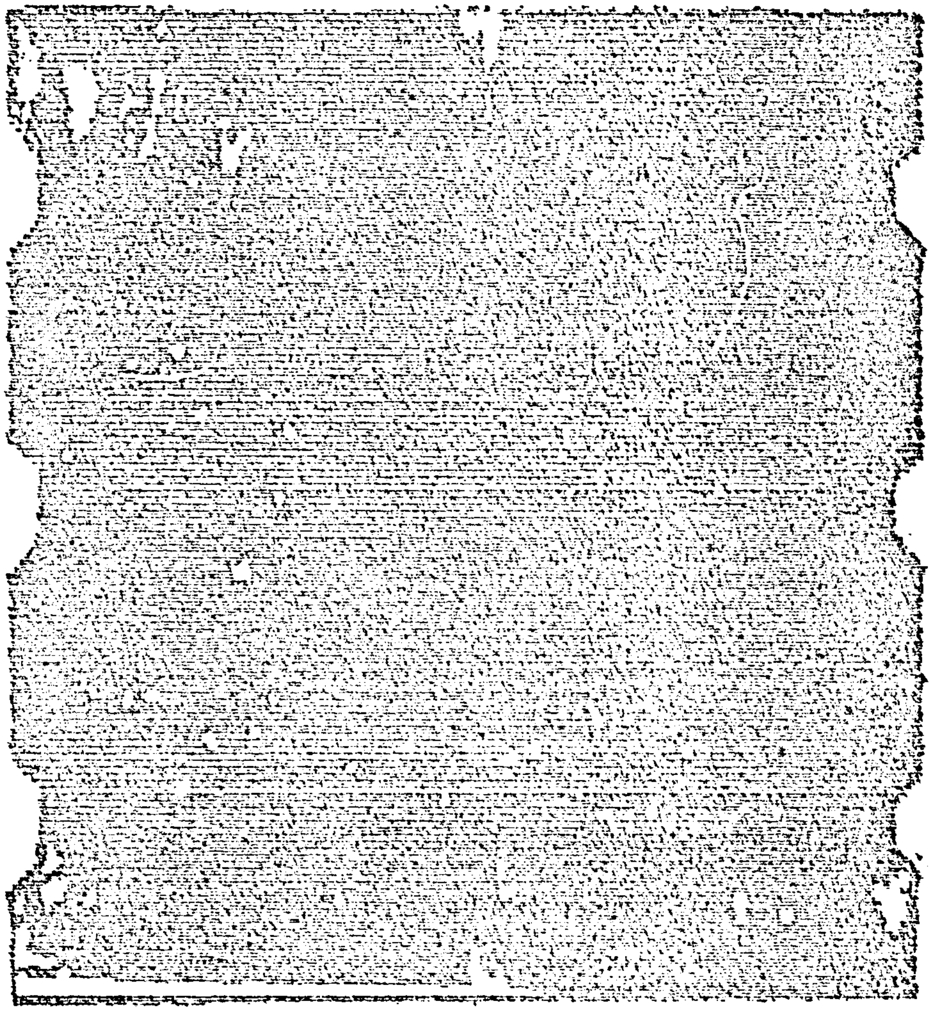

Figure 11. C-Scan of Laminate Imidized at 177 C, 150 Minutes

350/180 "A" 6/2/80

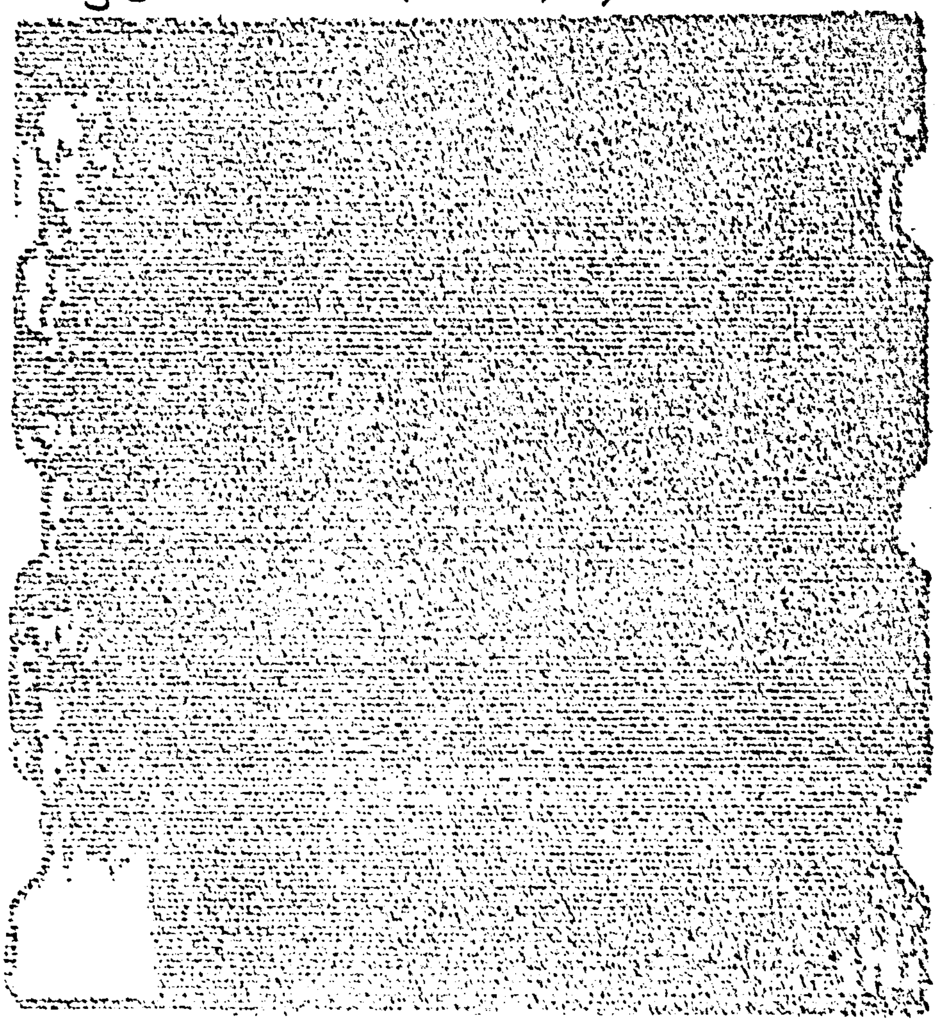

Figure 12. C-Scan of Laminate Imidized at 177 C, 180 Minutes

0375/30 'A' 6/2/80

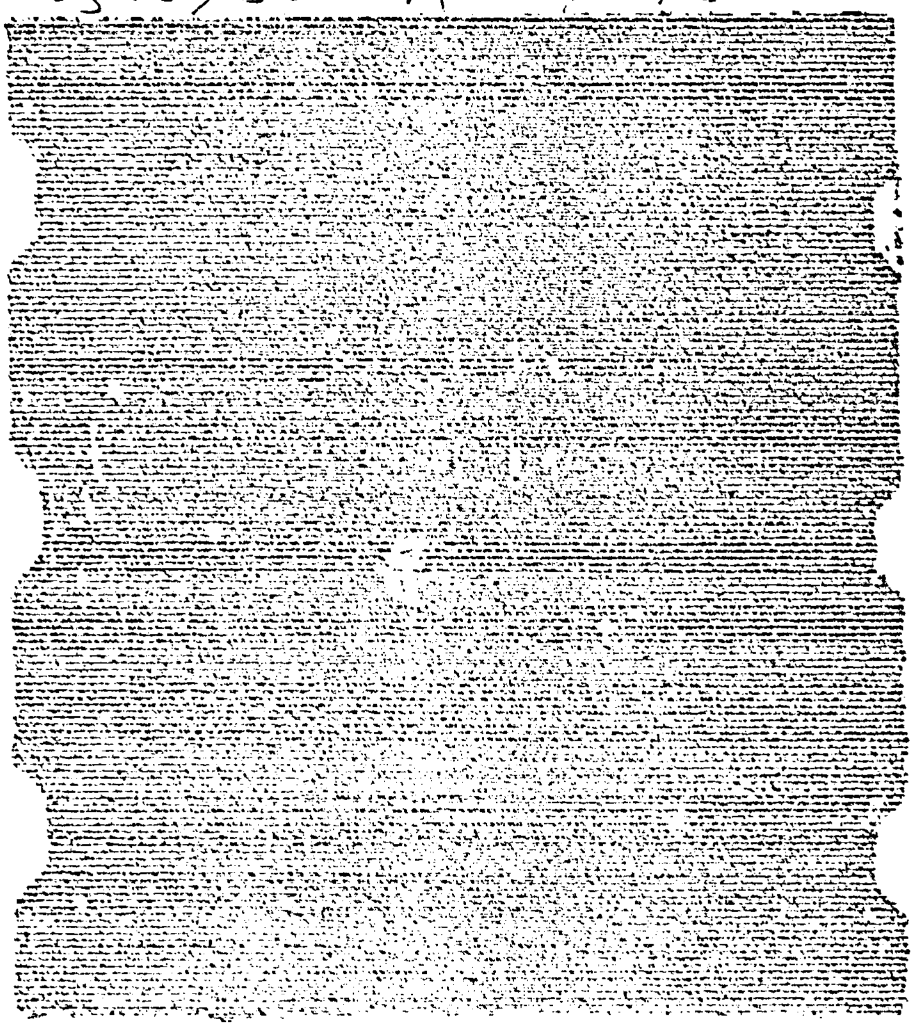

Figure 13. C-Scan of Laminate Imidized at 191 C, 30 Minutes

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375/60 "H" 6/2/80

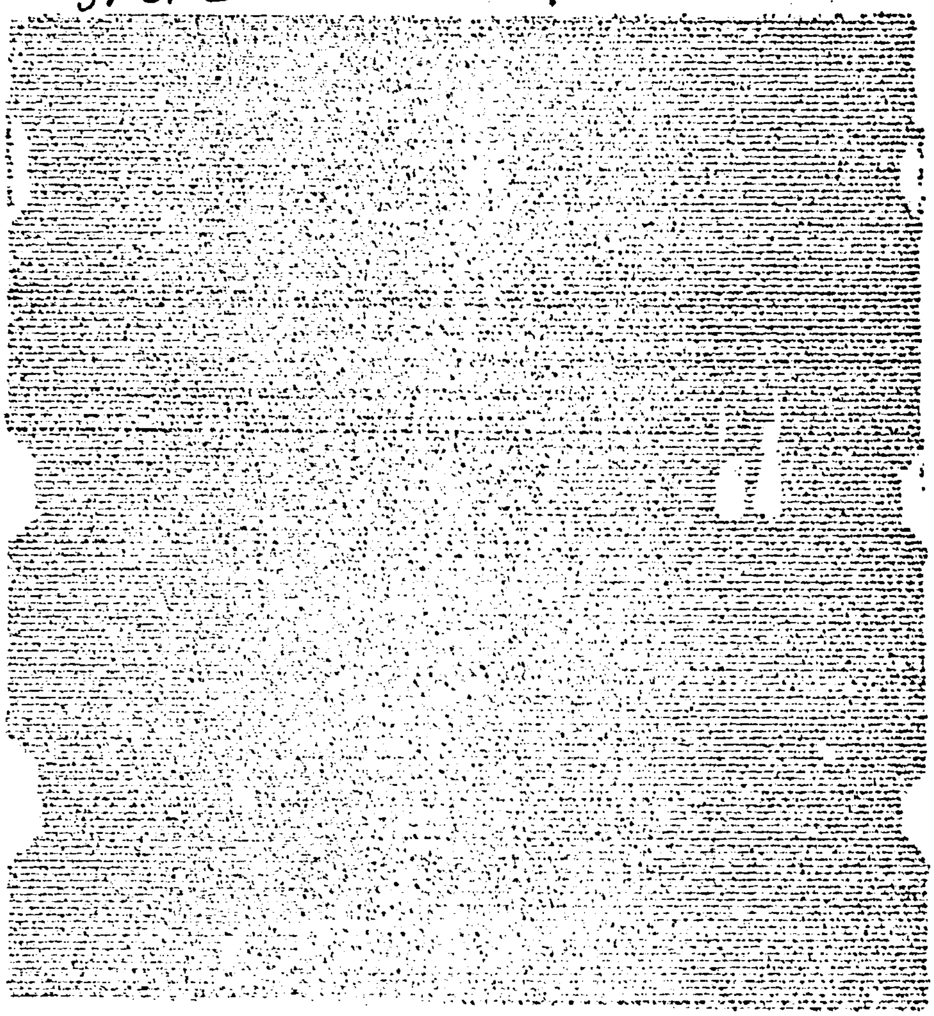


Figure 14. C-Scan of Laminate Imidized at 191 C, 60 Minutes

375/90 "A" 6/2/80

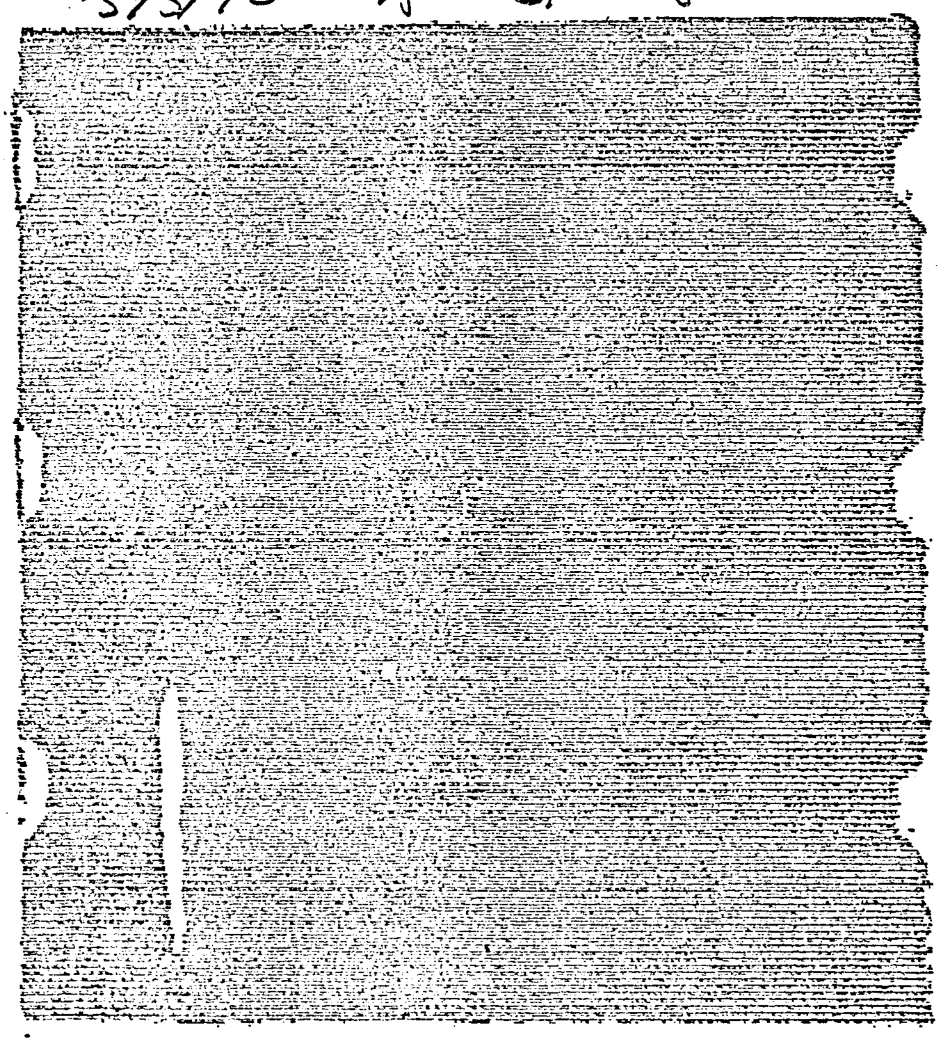


Figure 15. C-Scan of Laminate Imidized at 191 C, 90 Minutes

375/120 "A" 6/2/80

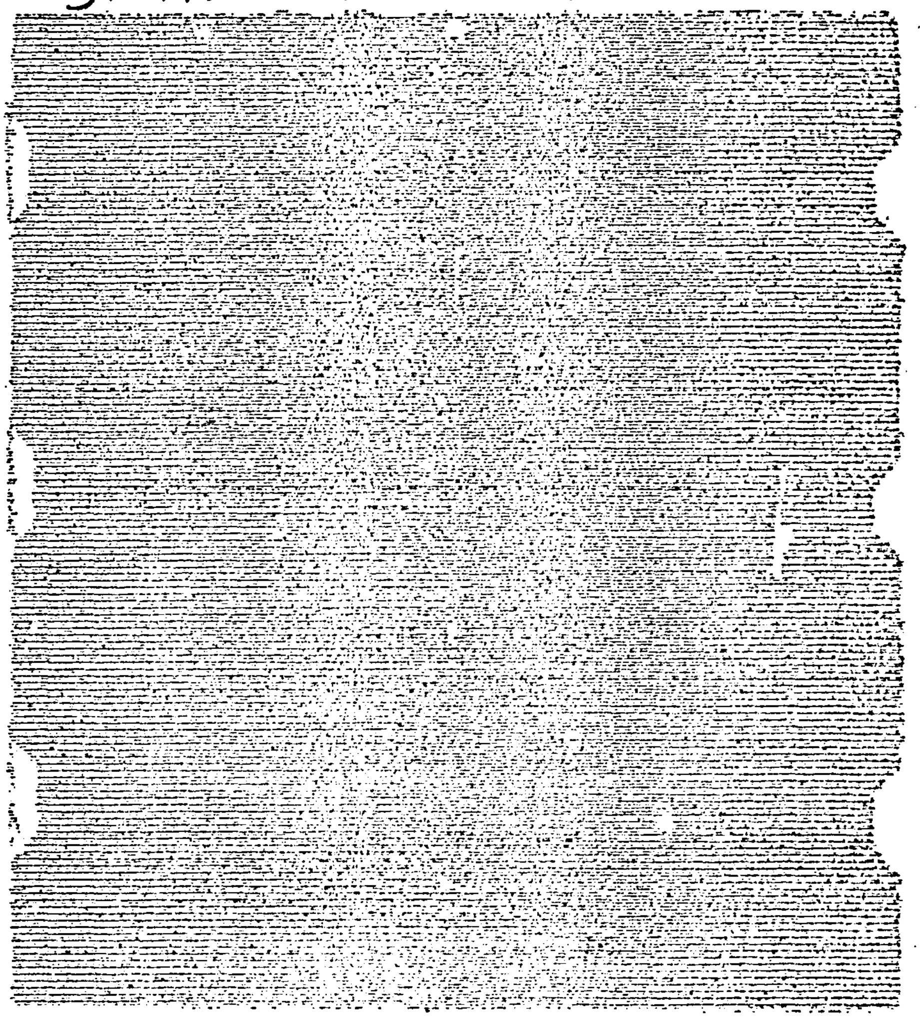


Figure 16. C-Scan of Laminate Imidized at 191 C, 120 Minutes

375/160 "A" 6/2/80

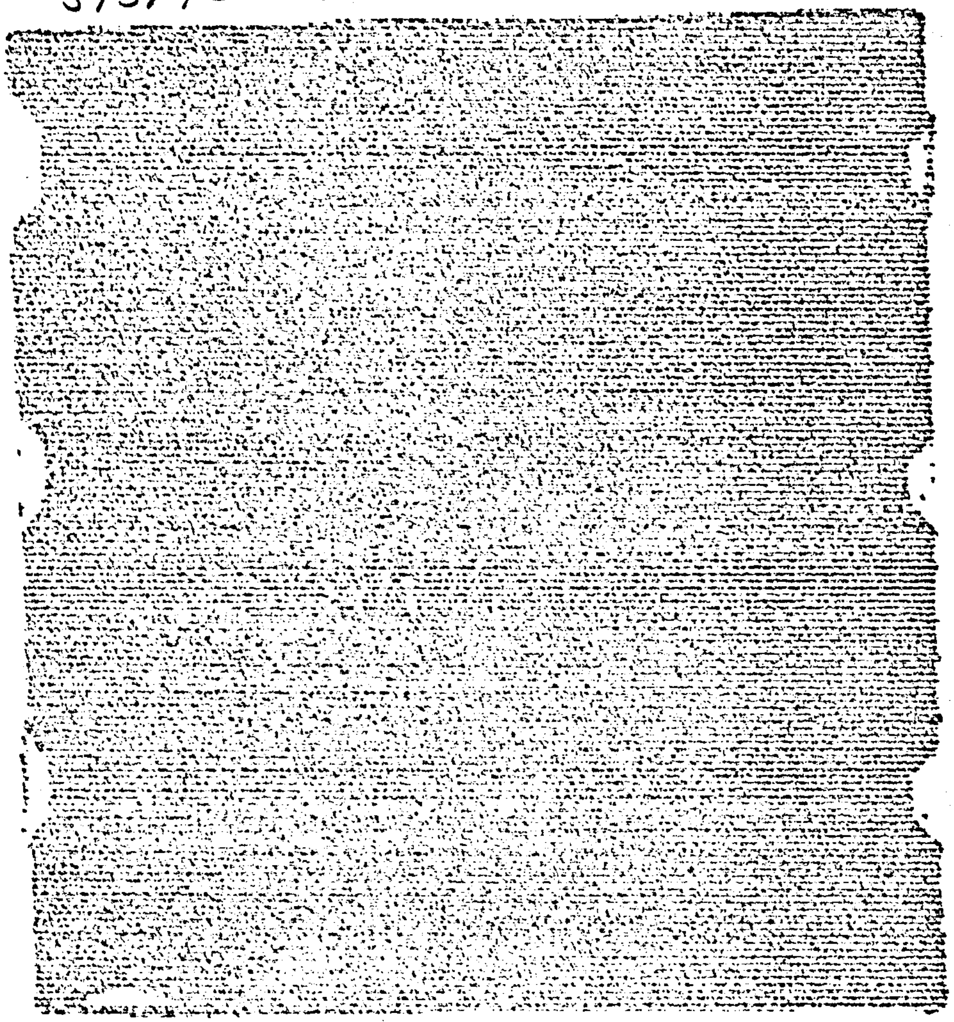


Figure 17. C-Scan of Laminate Imidized at 191 C, 150 Minutes

390/30 "A" 6/2/80

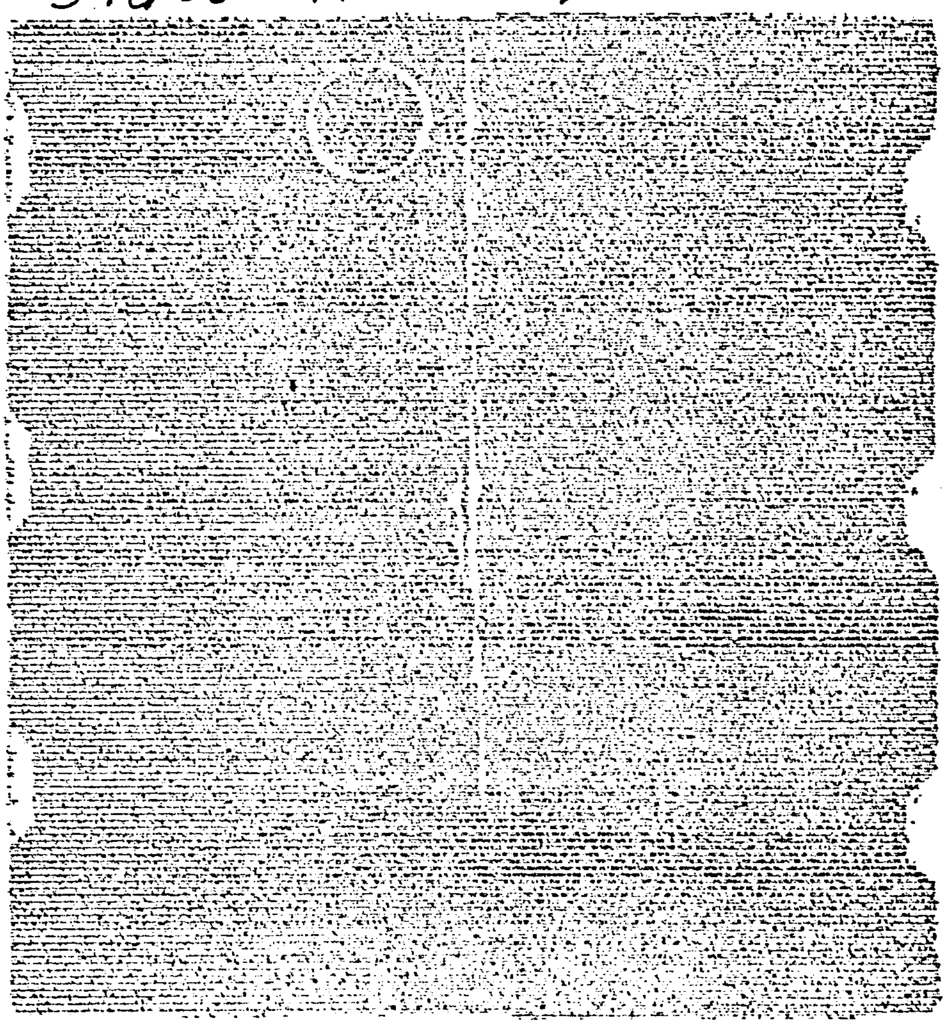


Figure 18. C-Scan of Laminate Imidized at 199 C, 30 Minutes

890/60 "A" 6/2/80

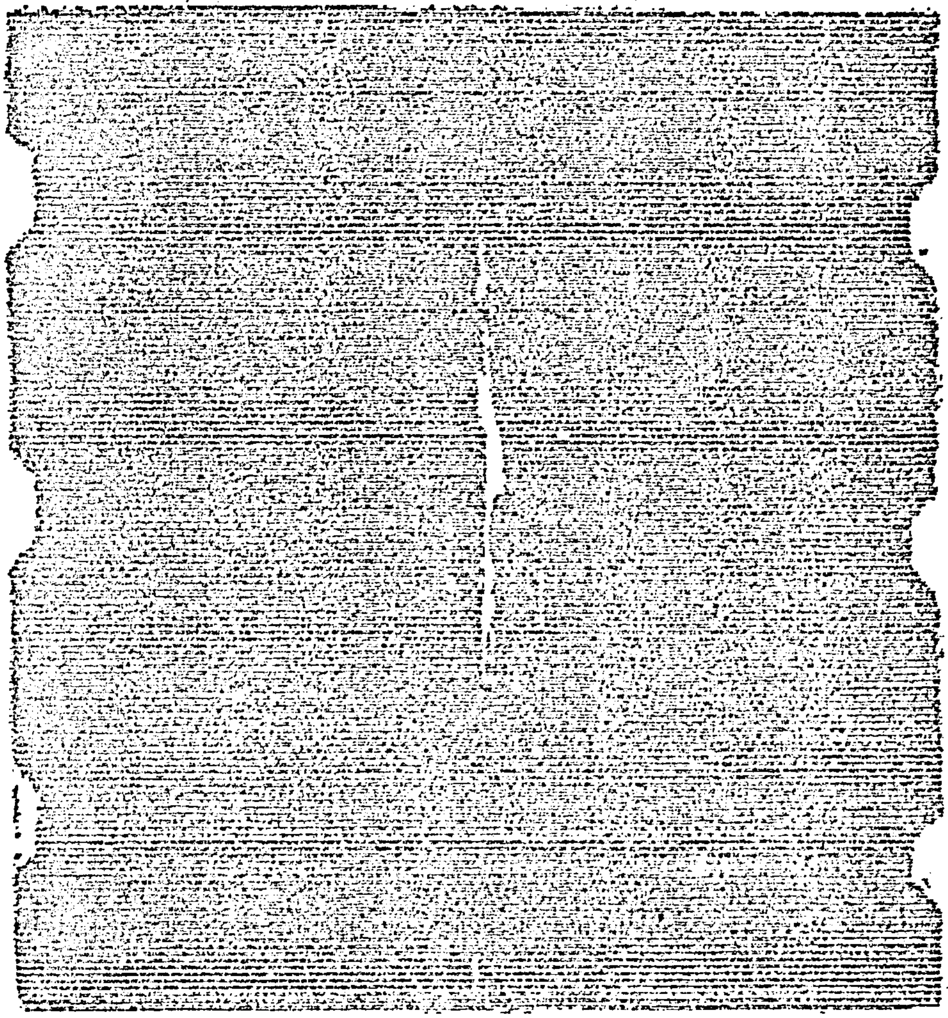


Figure 19. C-Scan of Laminate Imidized at 199 C, 60 Minutes

390/90 "A" 6/2/80

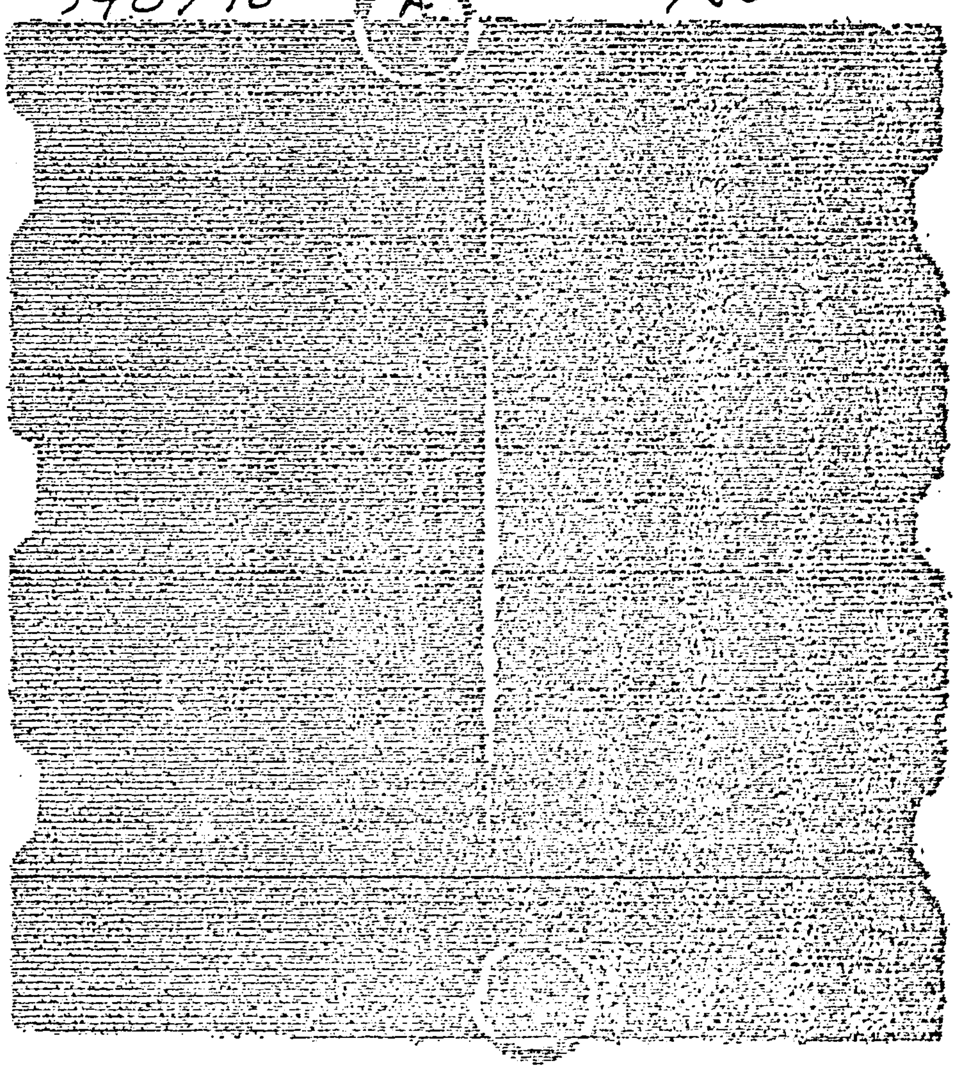


Figure 20. C-Scan of Laminate Imidized at 199 C, 90 Minutes

390/120 "A" 6/2/80

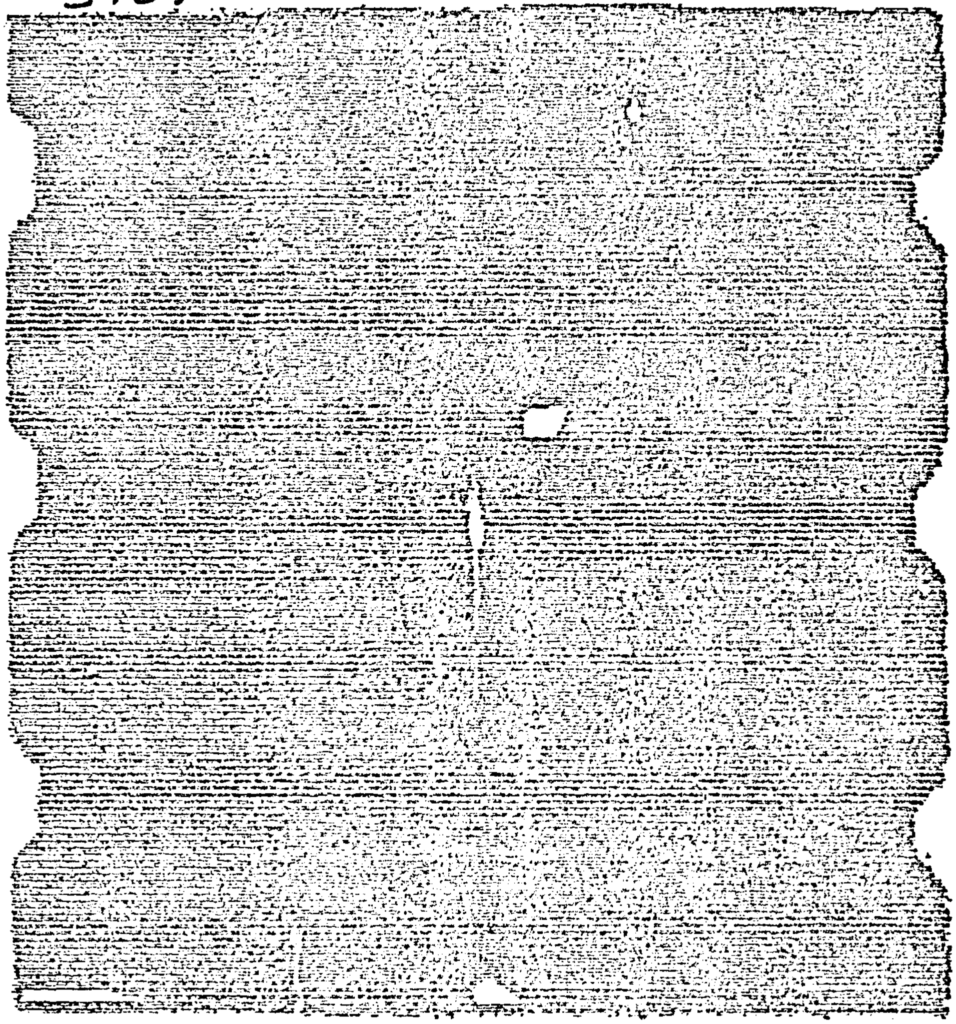


Figure 21. C-Scan of Laminate Imidized at 199 C, 120 Minutes

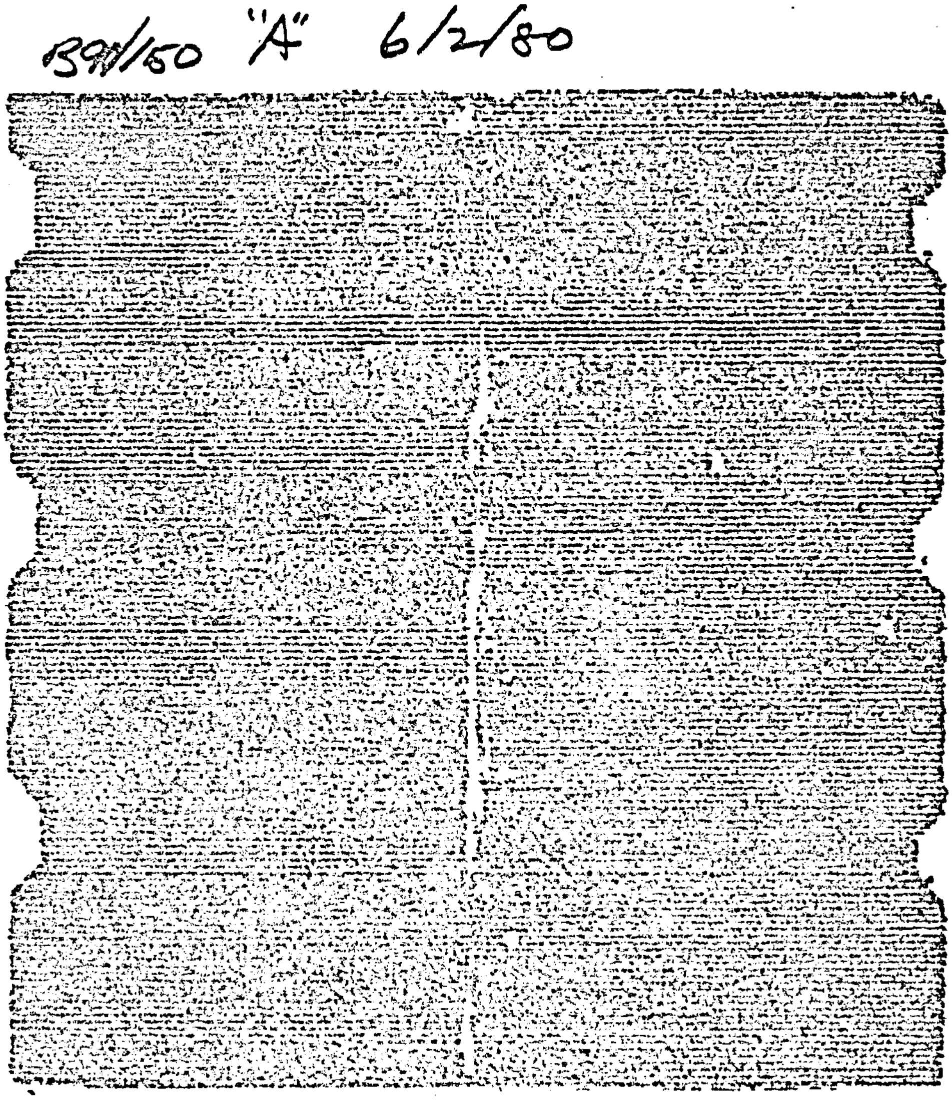


Figure 22. C-Scan of Laminate Imidized at 199 C, 150 Minutes

425/30/ "A" 6/2/80

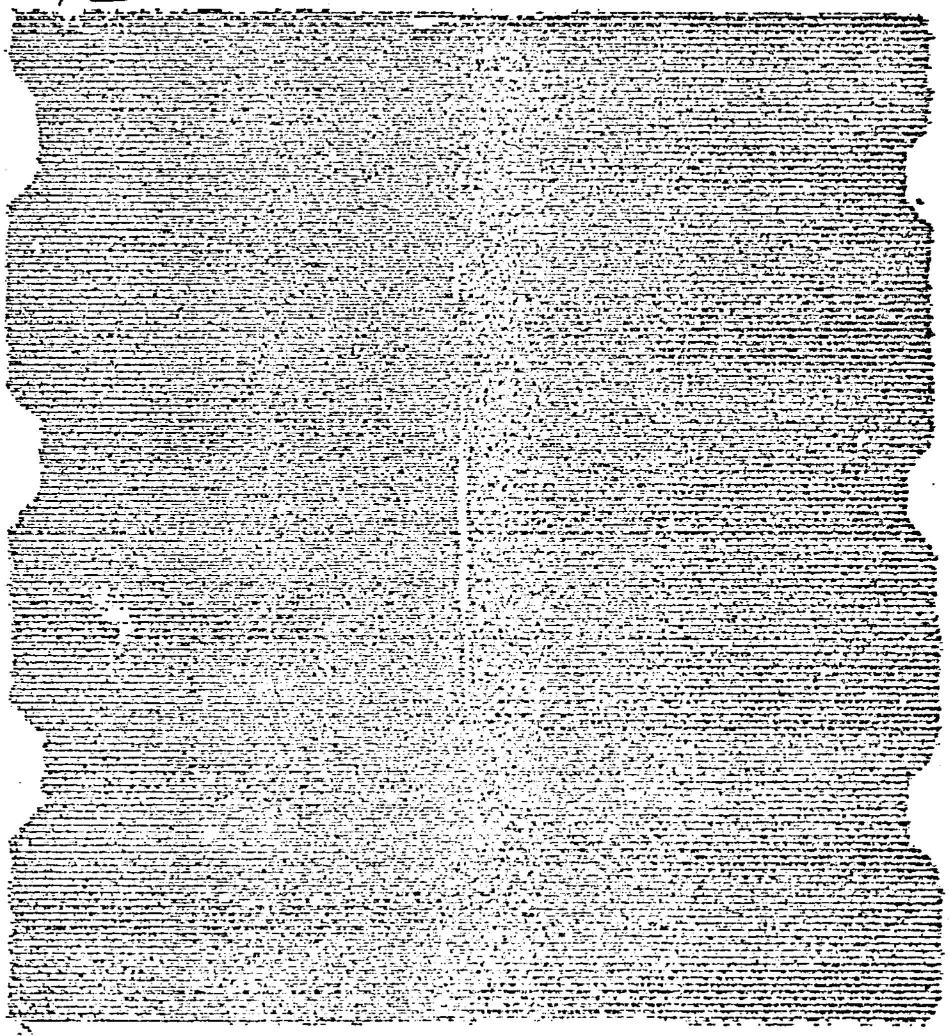


Figure 23. C-Scan of Laminate Imidized at 218 C, 30 Minutes

4-25/60 "A" 6/2/80

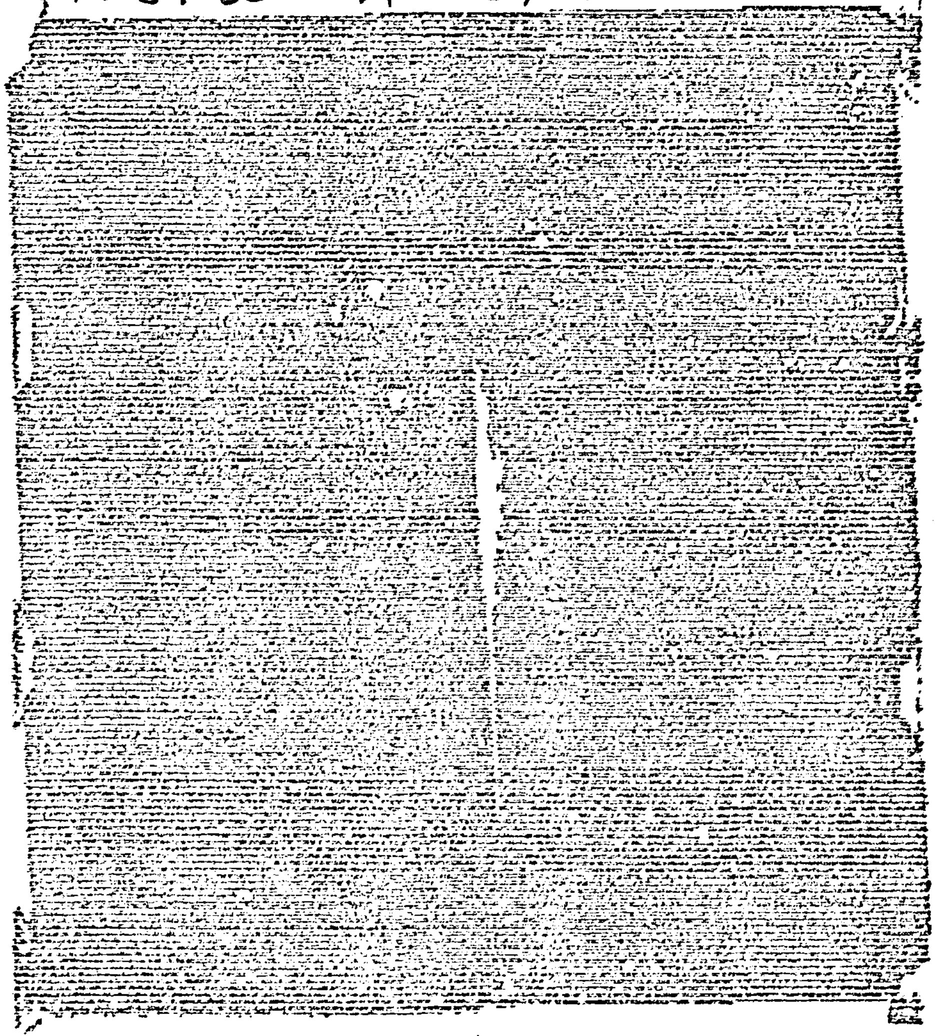


Figure 24. C-Scan of Laminate Imidized at 218 C, 60 Minutes

425/90 "A" 6/2/80

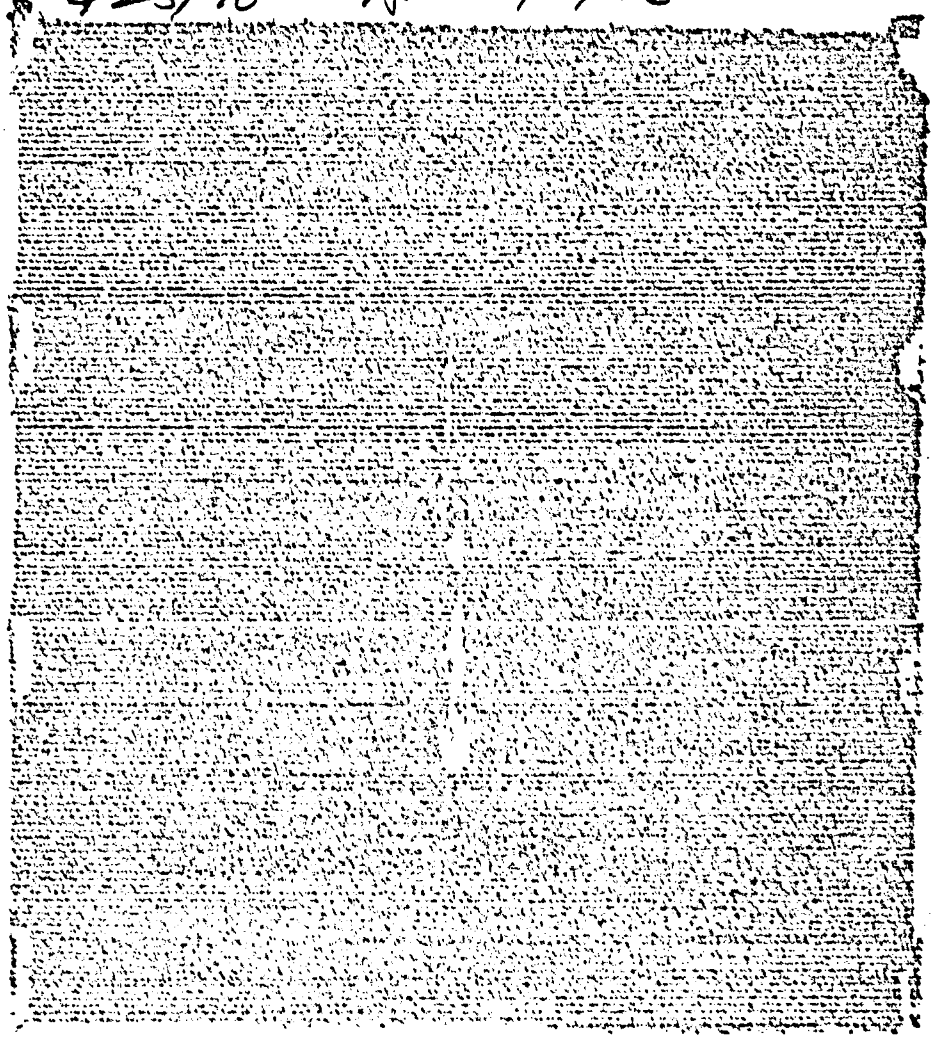


Figure 25. C-Scan of Laminate Imidized at 218 C, 90 Minutes

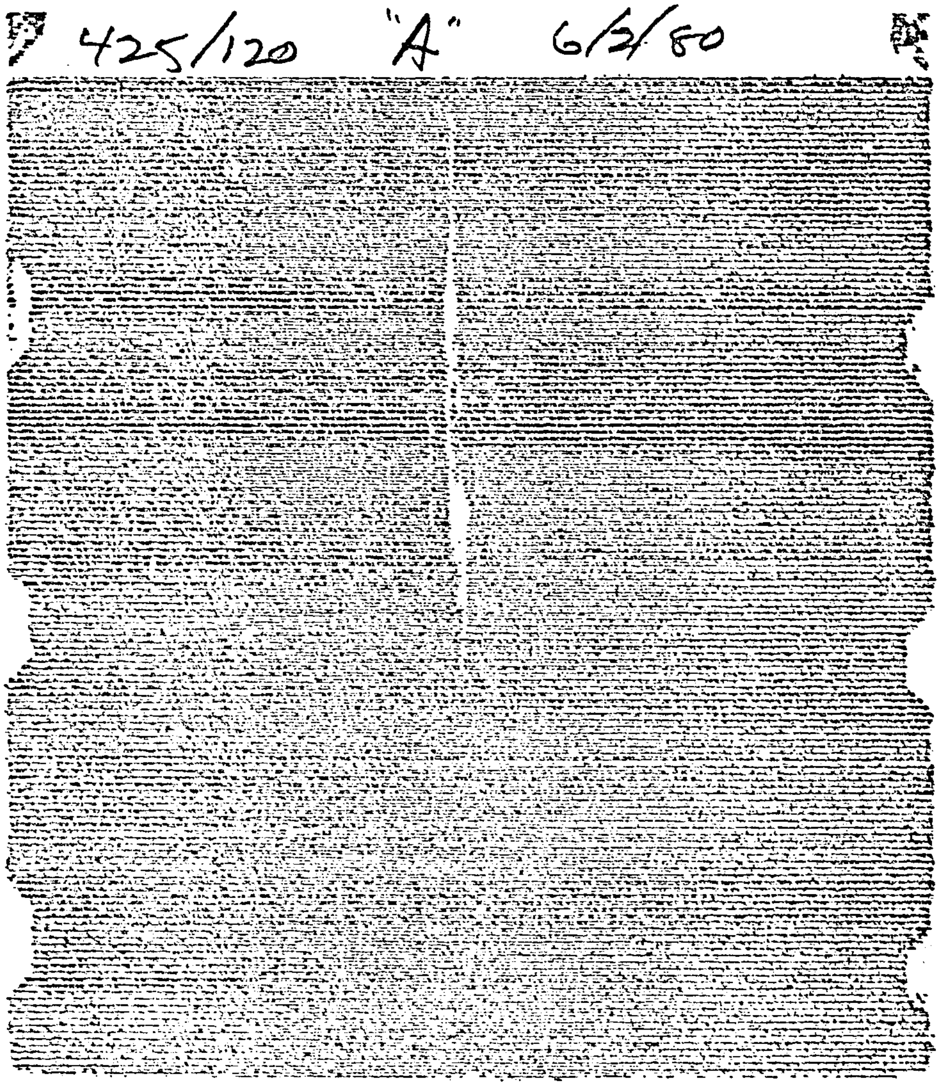


Figure 26. C-Scan of Laminate Imidized at 218 C, 120 Minutes

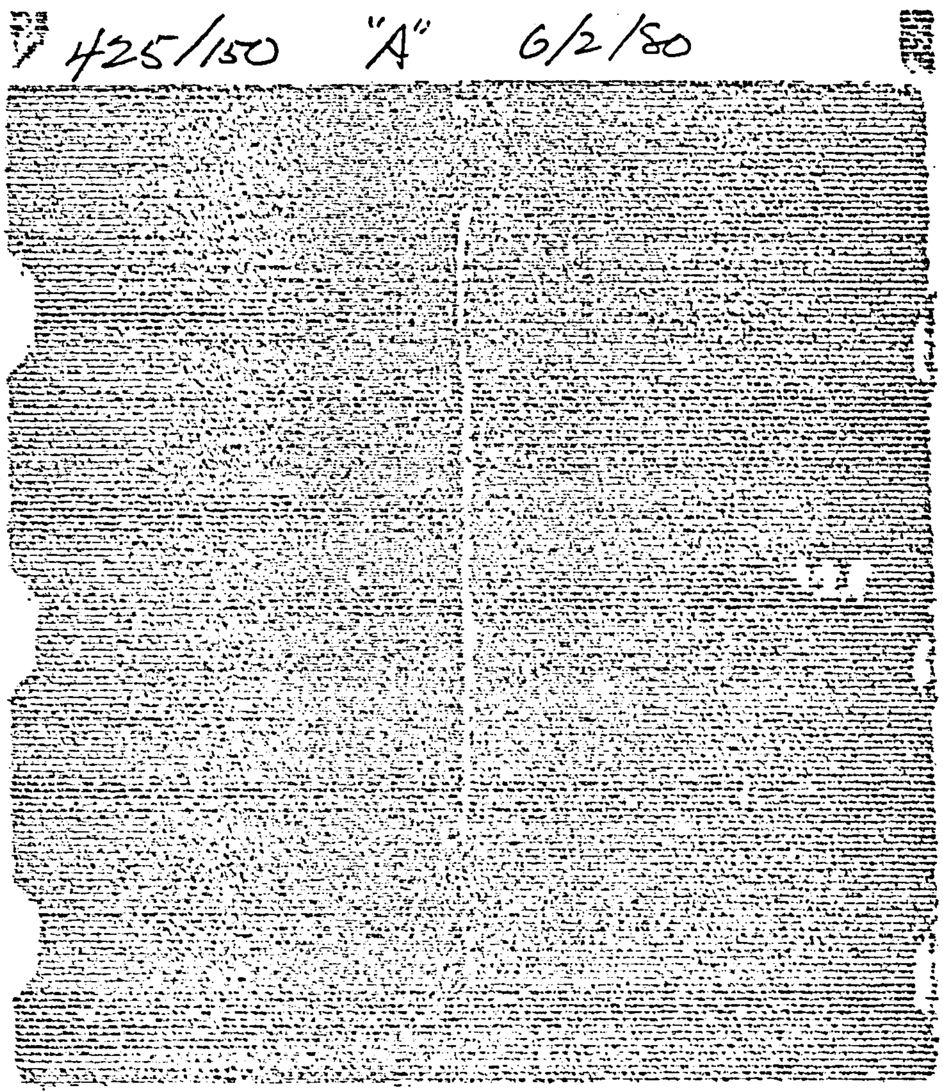
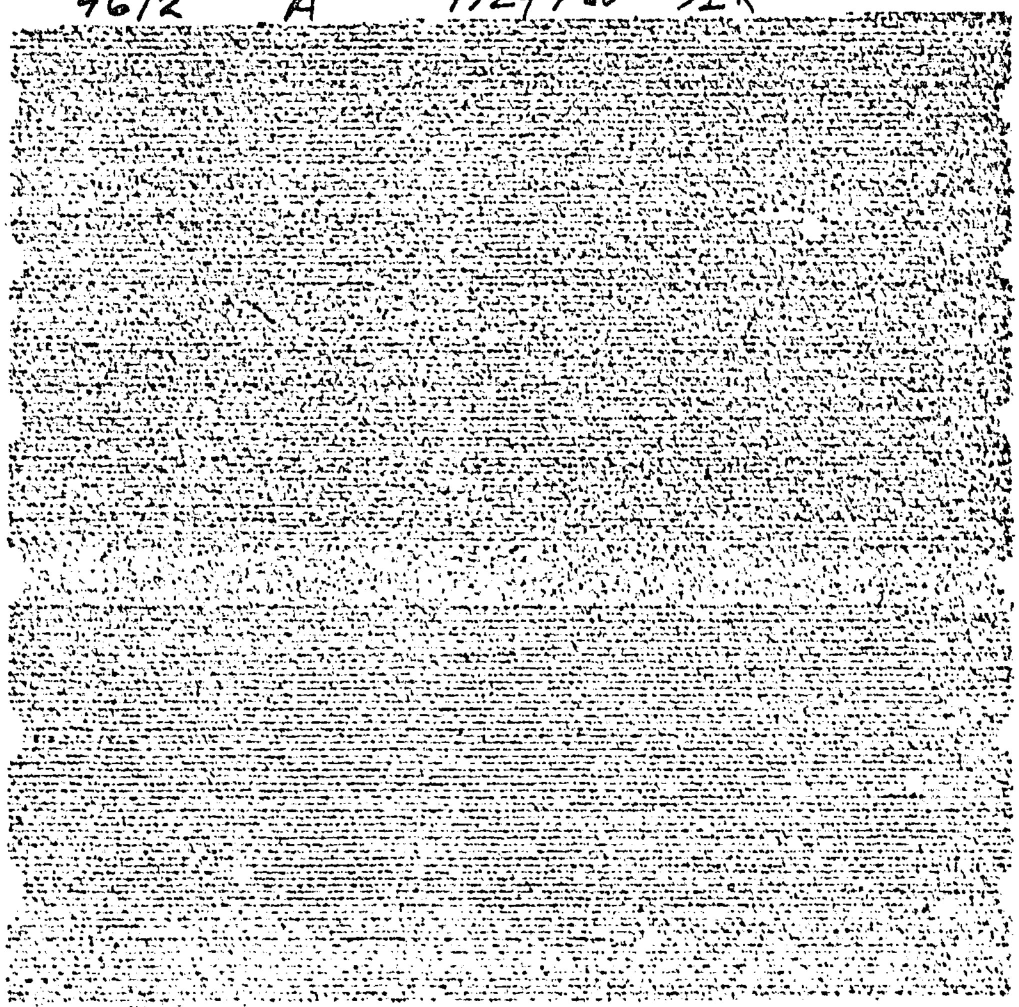


Figure 27. C-Scan of Laminate Imidized at 218 C, 150 Minutes

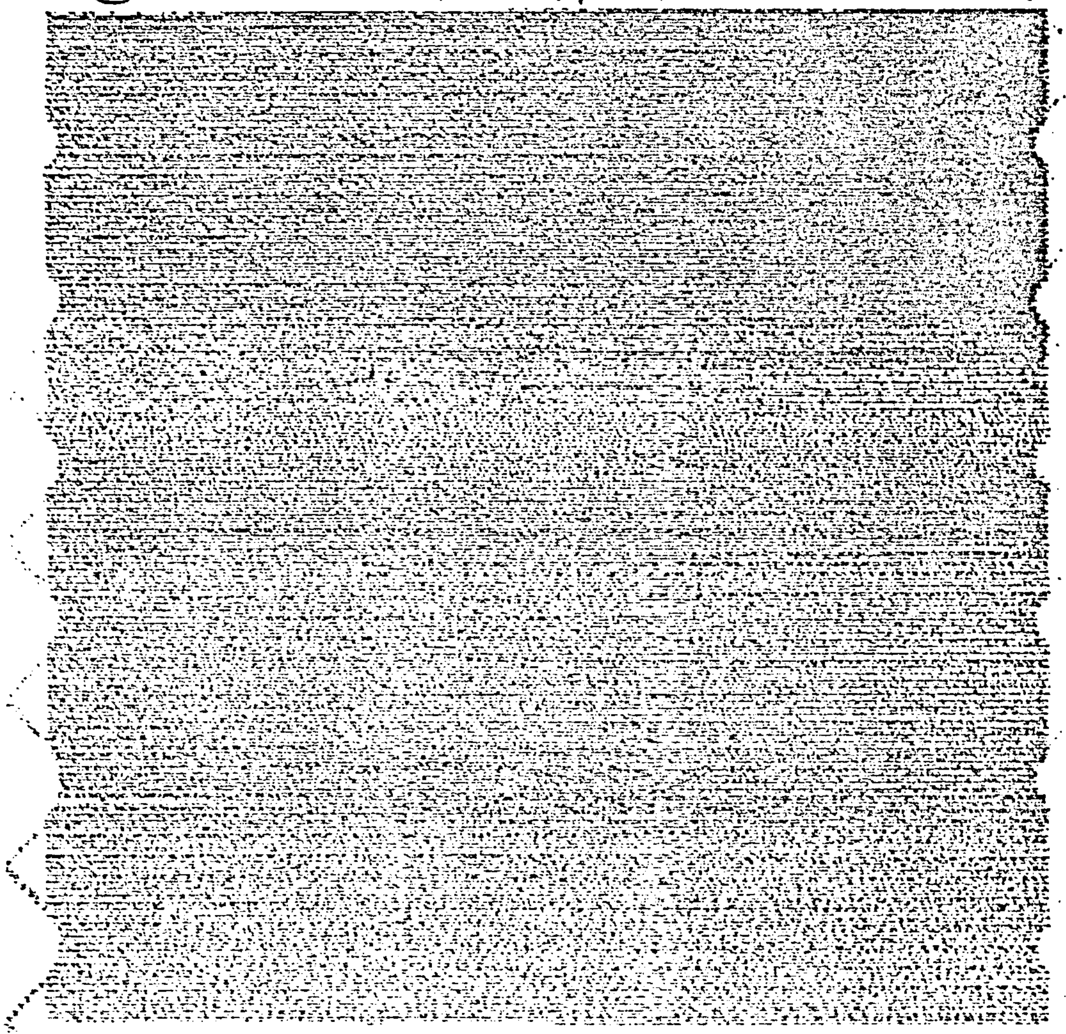
4612 'A' 4/29/80 1/2X



STANDARD CELION 3000 (EPOXY RESIN SIZED)/LARC 160 PREPREG

Figure 28. C-Scan of Process Verification Laminate, Prepreg Batch U.S.P 2W4612

C0161 "A" 4/29/80 1/2X



STANDARD CELION/LARC 160 PREPREG

Figure 29. C-Scan of Process Verification Laminate, Prepreg Batch Fiberite C0161

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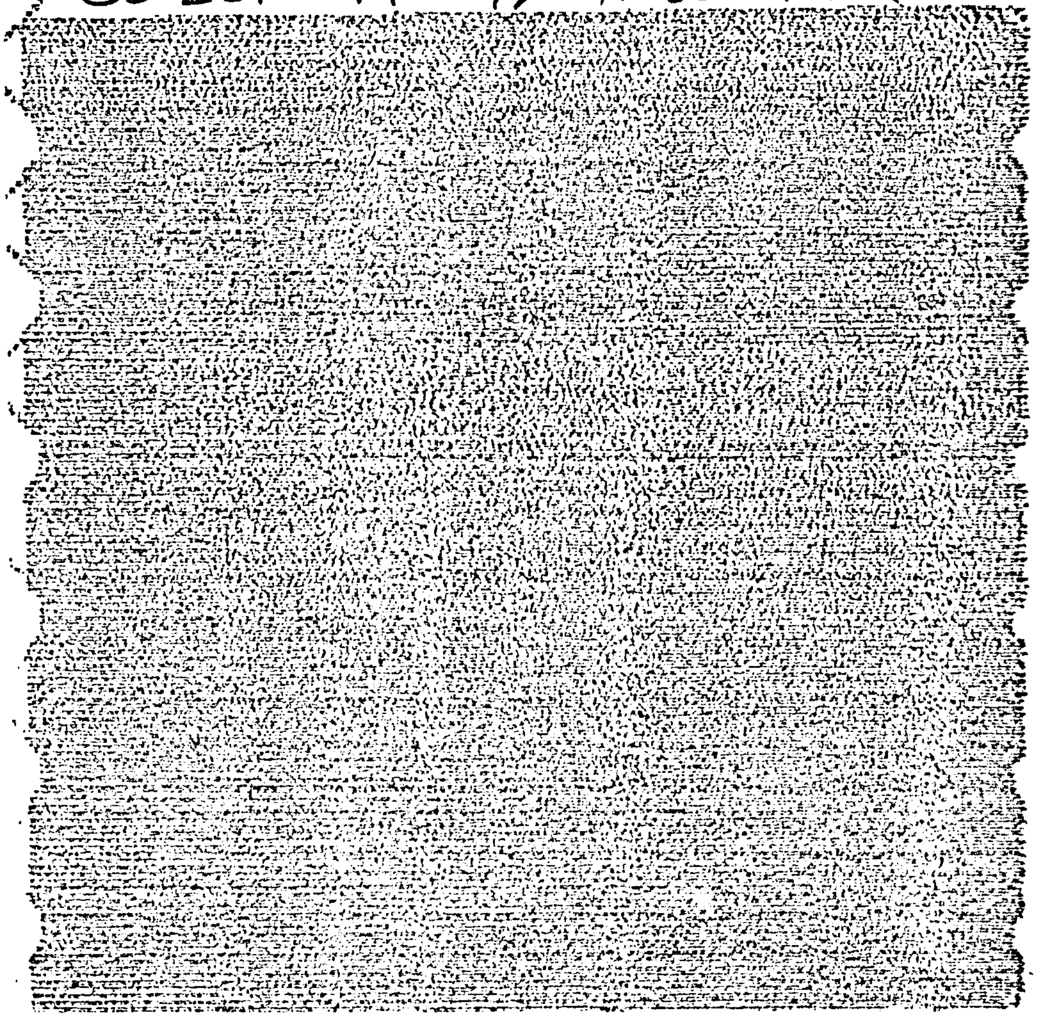
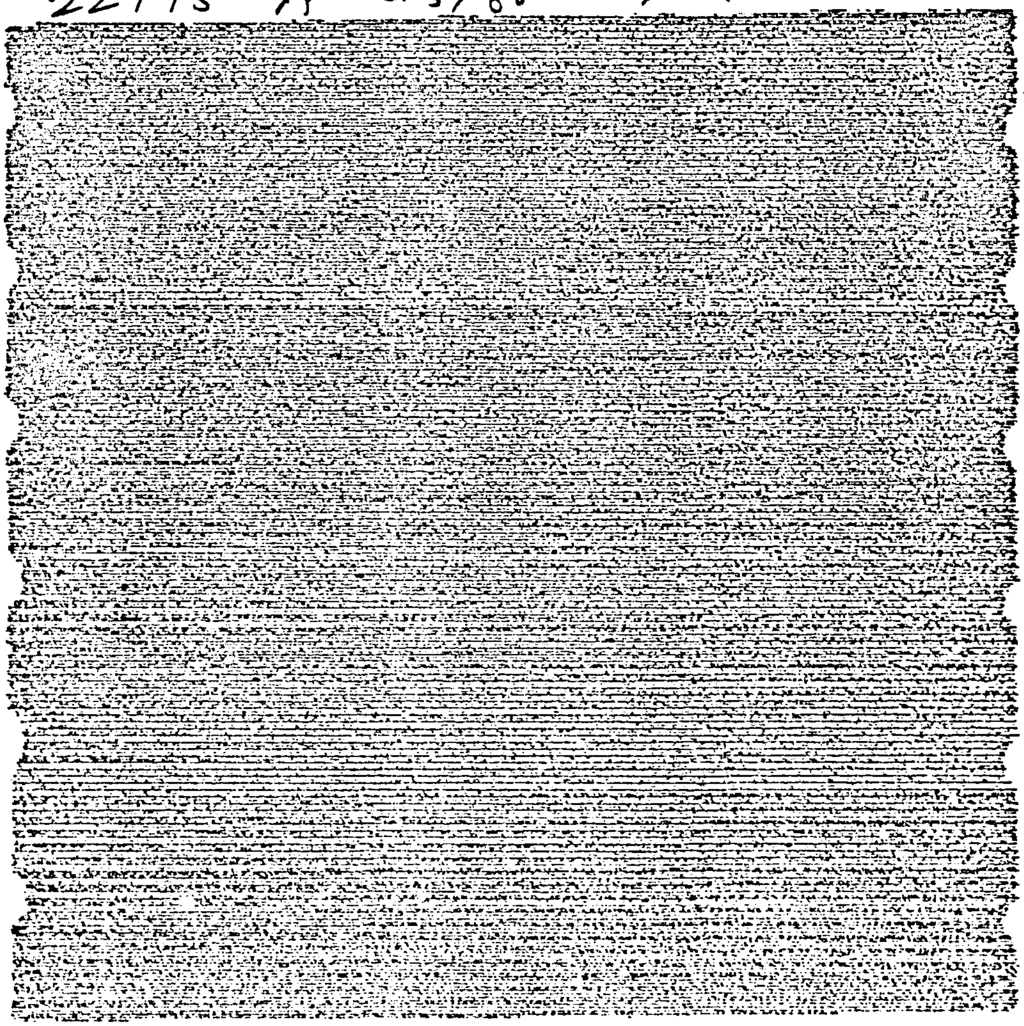
C0261 "A" 4/29/80 1/2X



STANDARD CELION/LARC 160 PREPREG

Figure 30. C-Scan of Process Verification Laminate, Prepreg Batch Fiberite C0261

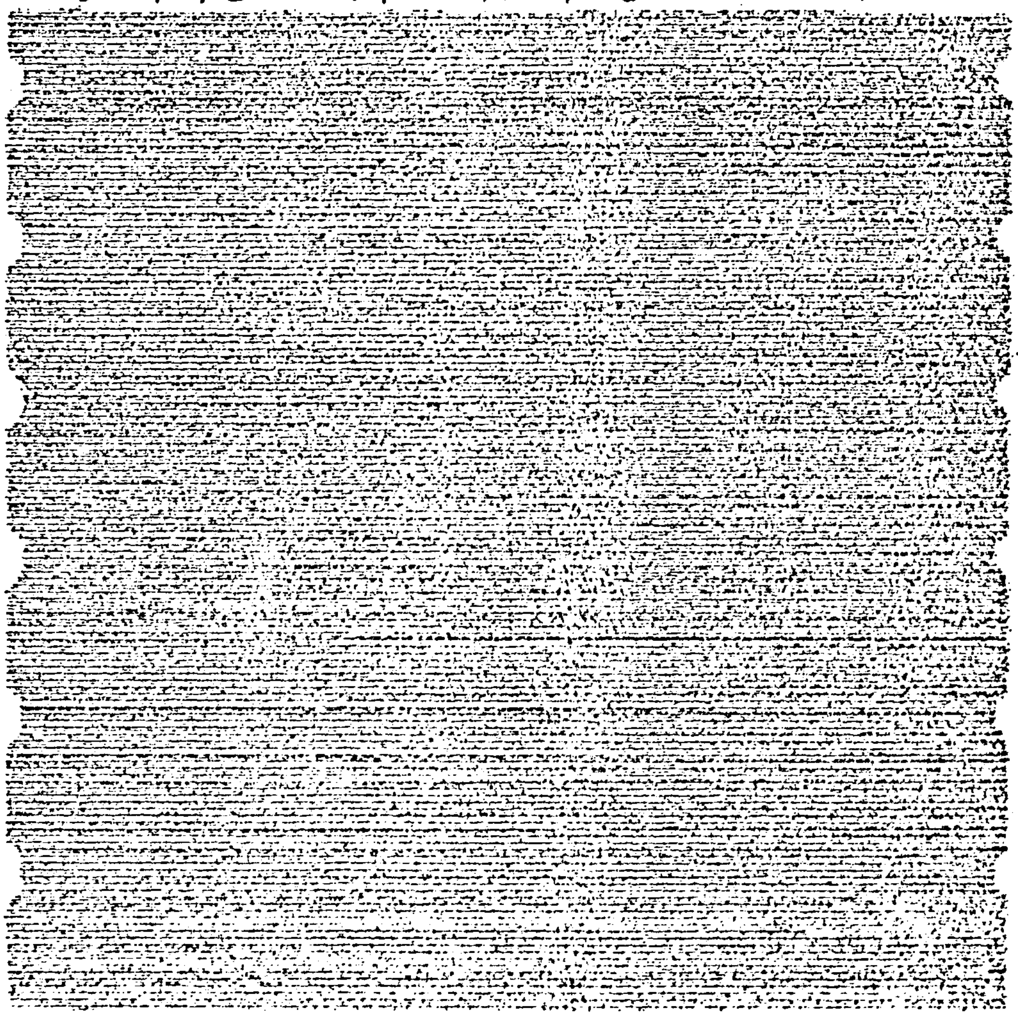
22945 "A" 5/5/80 1/2 X.



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
+5%	STD	STD	STD

Figure 31. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22945

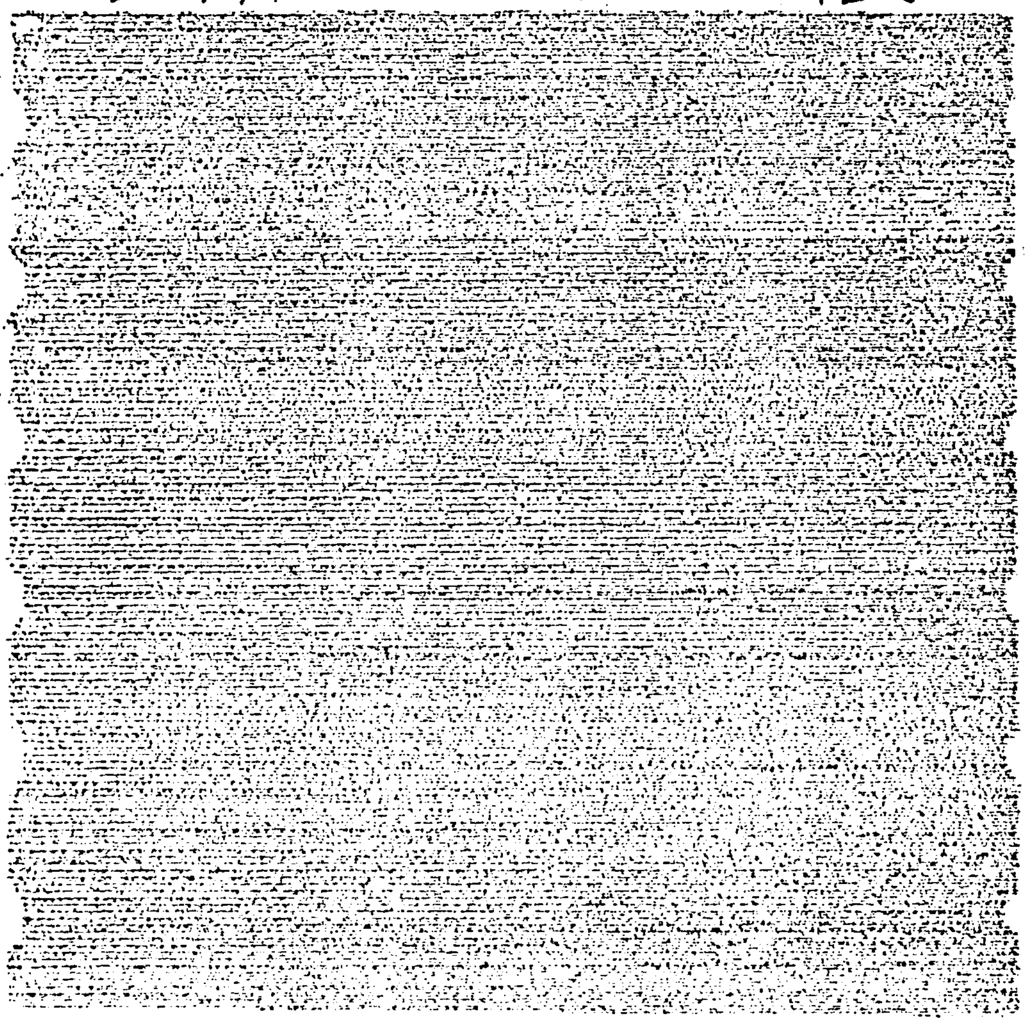
22946 "A" 4/29/80 1/2 X



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
-5%	STD	STD	STD

Figure 32. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22946

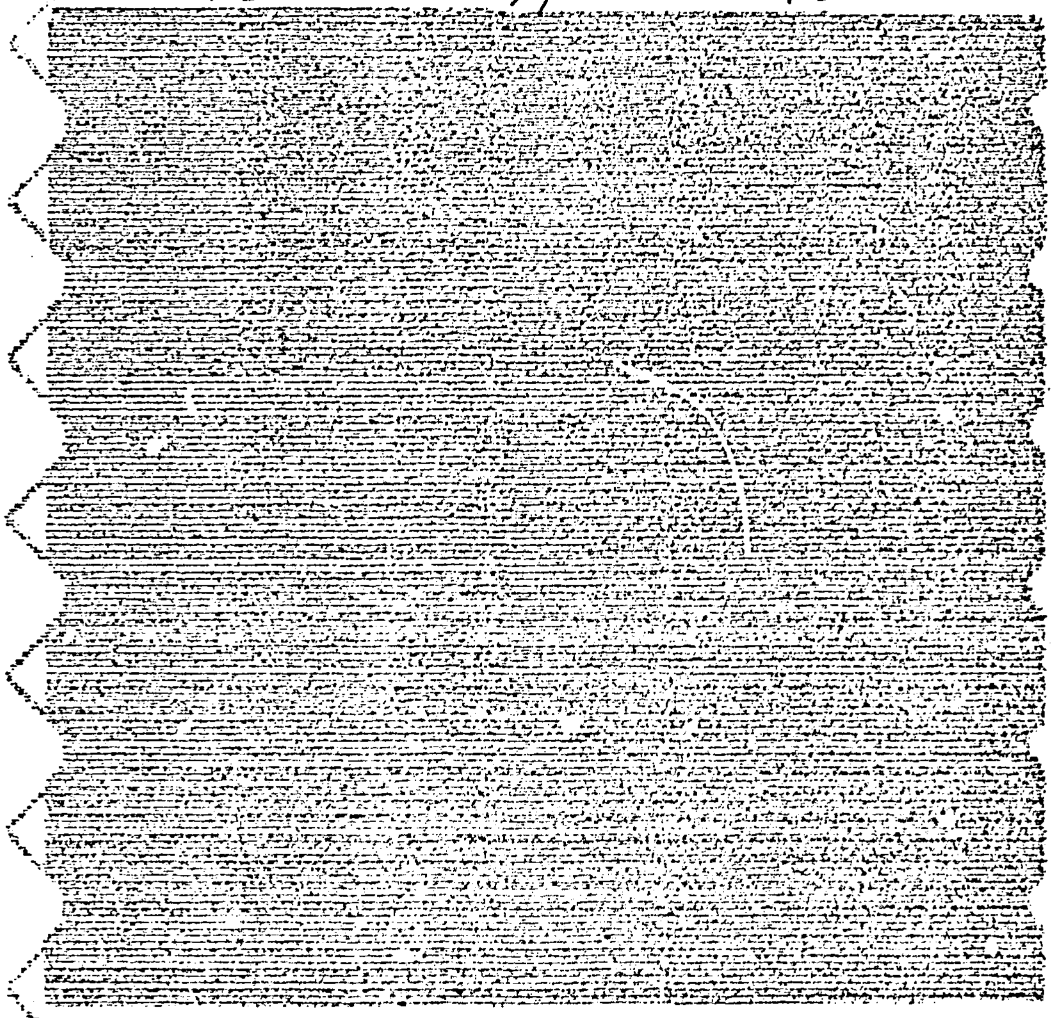
22949 "A" 4/29/80 1/2X



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	NA (+5%) BTDA (STD)	STD	STD

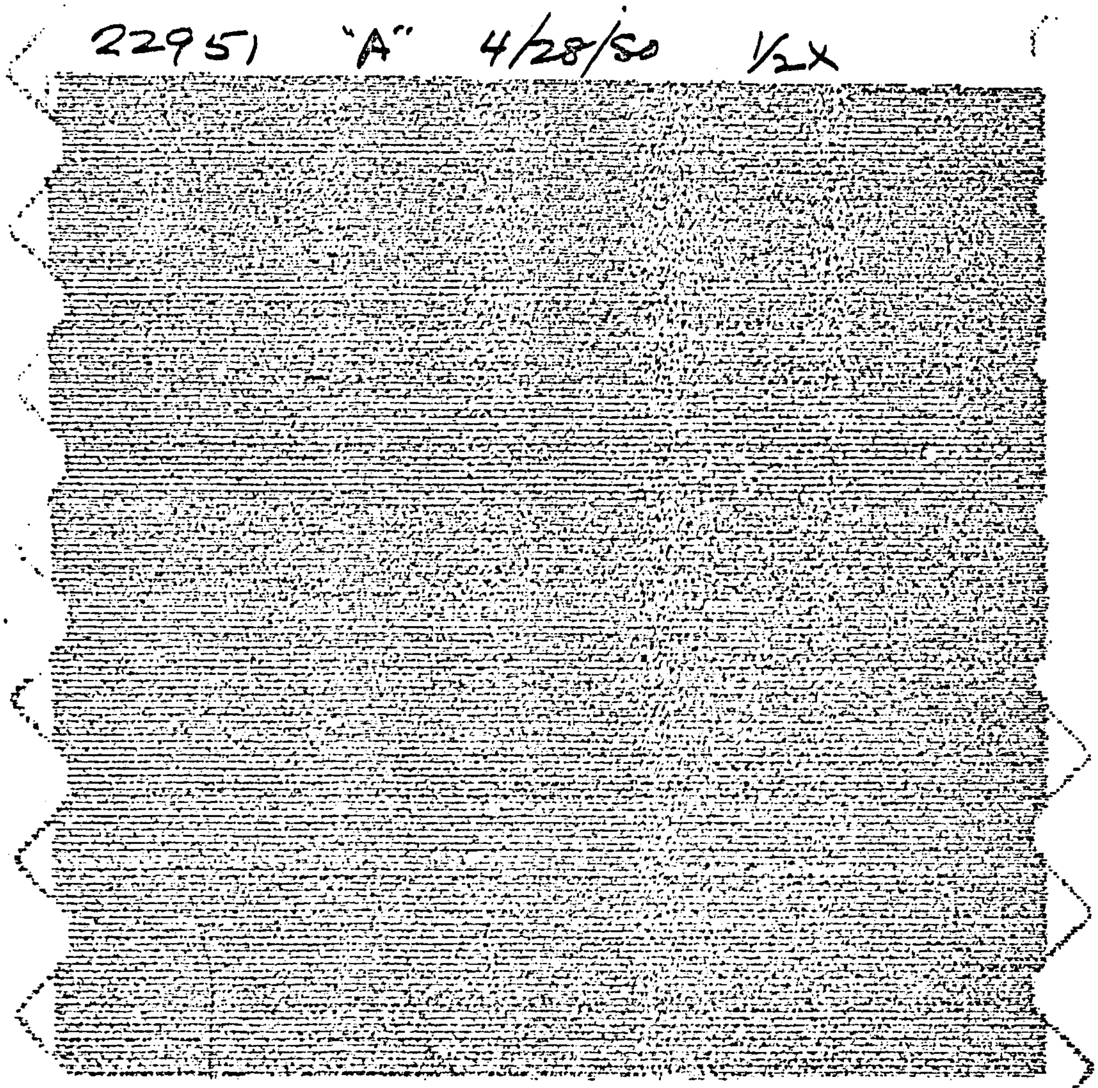
Figure 33. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22949

22950 "A" 4/29/80 1/2X



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	NA (-5%) BTDA (STD)	STD	STD

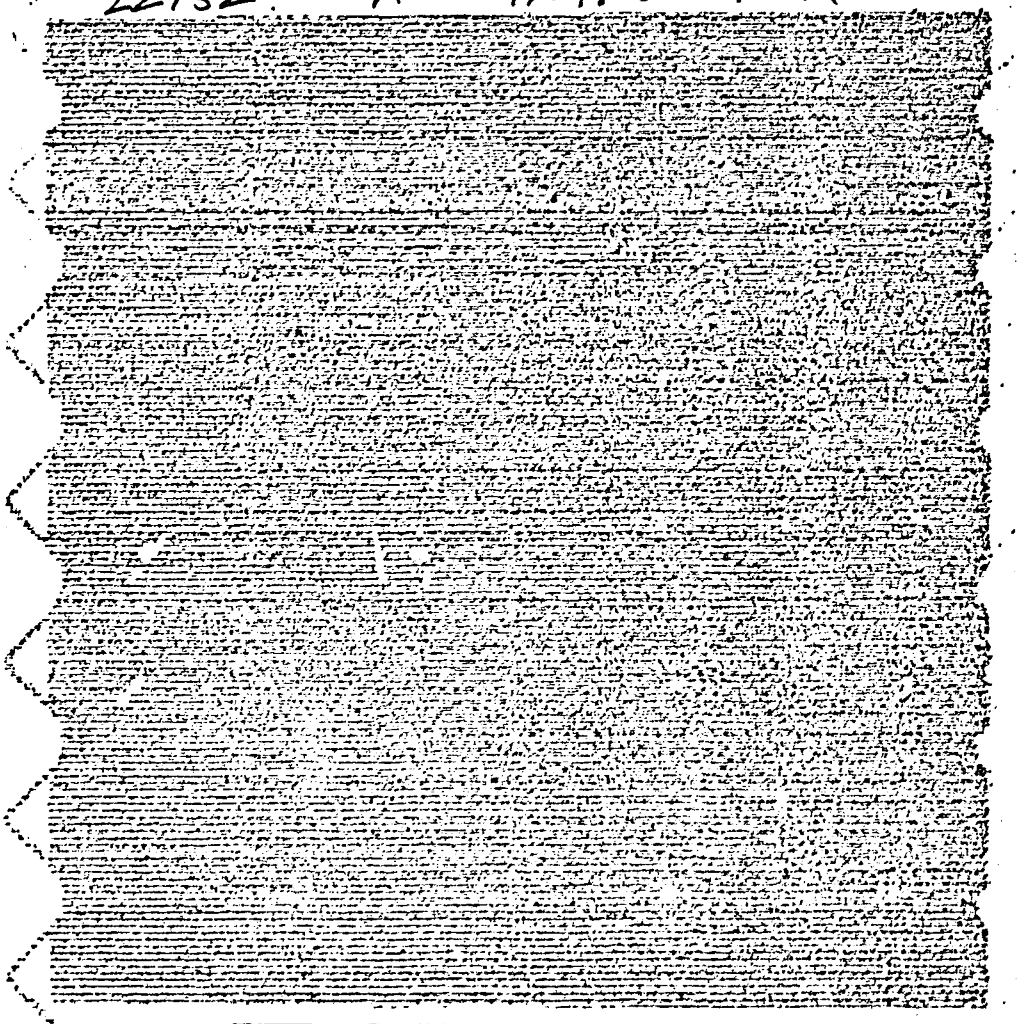
Figure 34. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22950



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	NA (STD) BTDA (+5%)	STD	STD

Figure 35. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22951

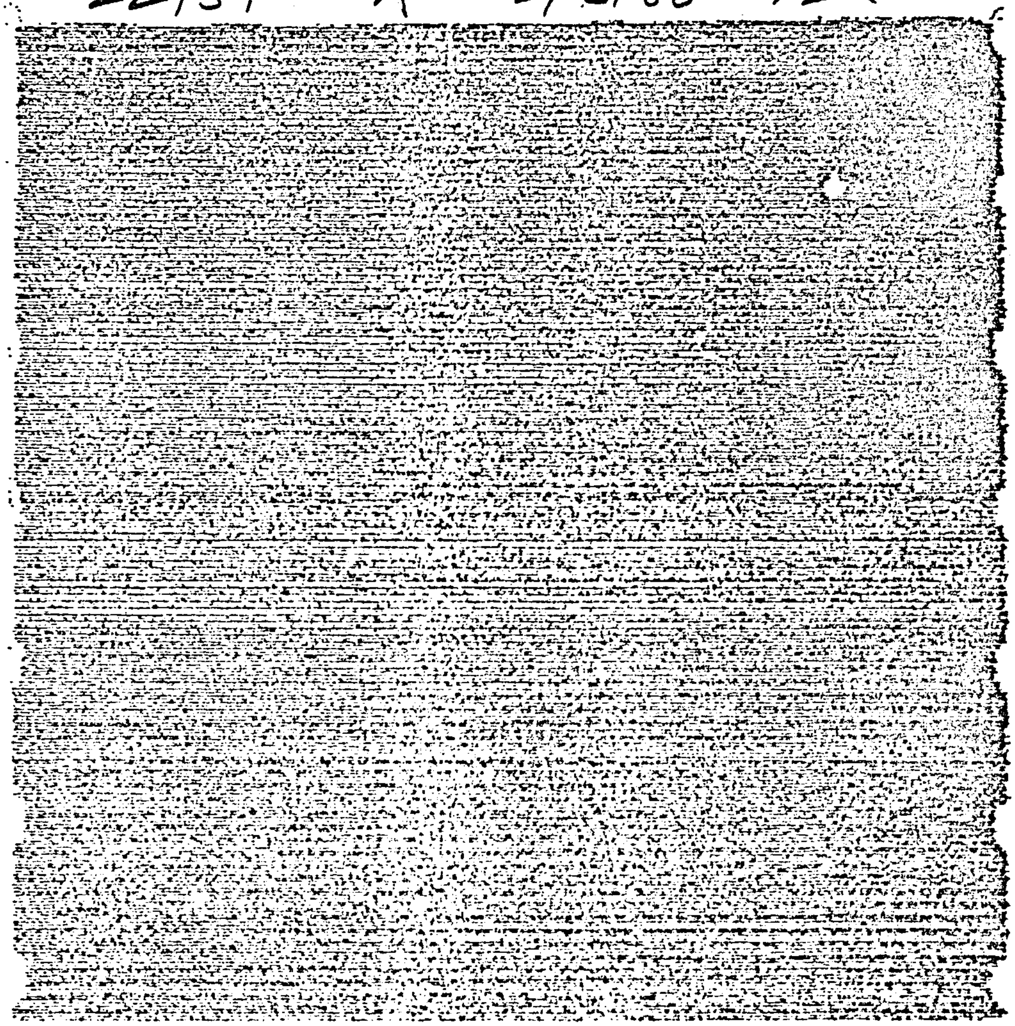
22952 "A" 4/29/80 1/2X



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	NA (STD) BTDA (-5%)	STD	STD

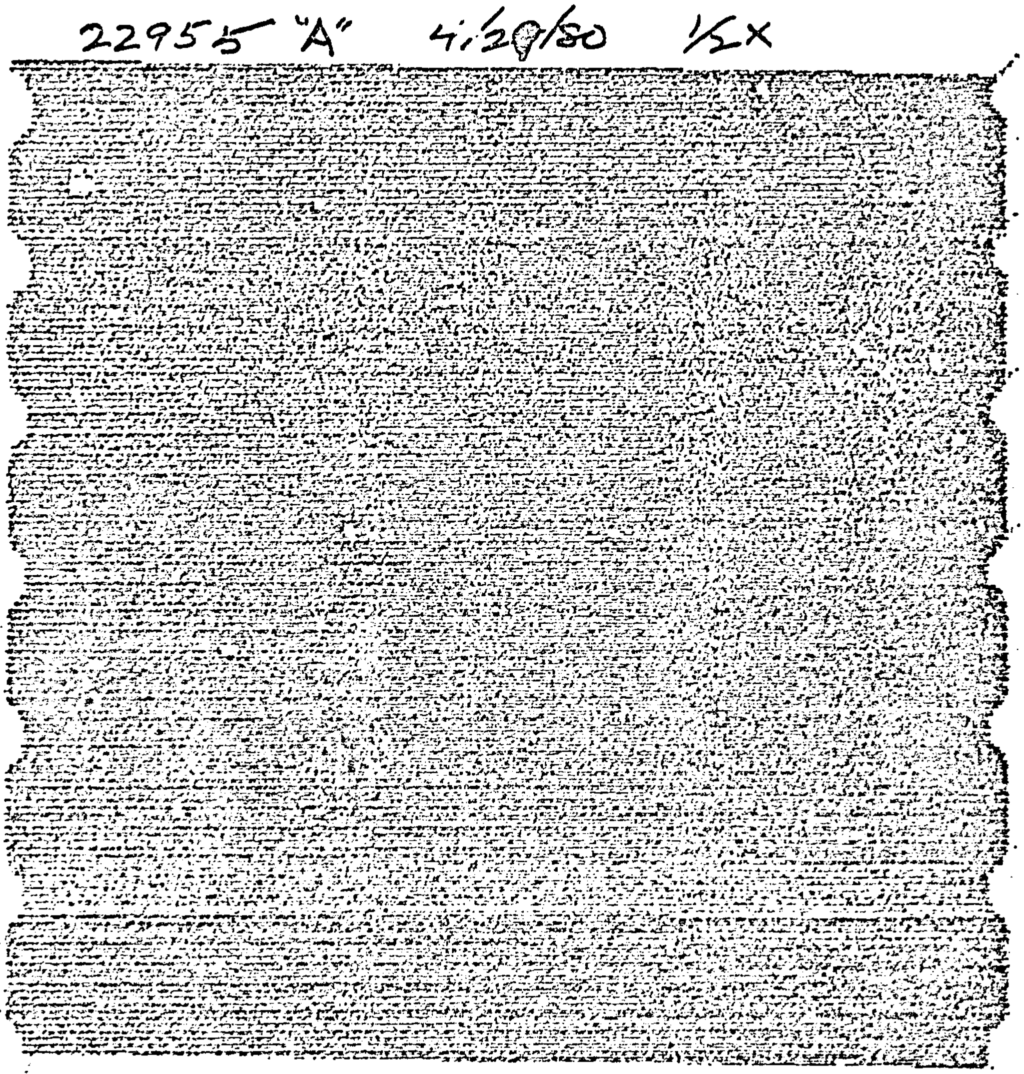
Figure 36. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22952

22954 "A" 5/5/80 1/2X



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	STD	2 HR AT 60 C	STD

Figure 37. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22954



RESIN FORMULATION VARIABLES		RESIN PROCESS VARIABLES	
CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
STD	STD	STD	6 HR

Figure 38. C-Scan of Process Verification Laminate, Prepreg Batch Hexcel 22955

EX 242 "A" 2/26/80 1/4 X

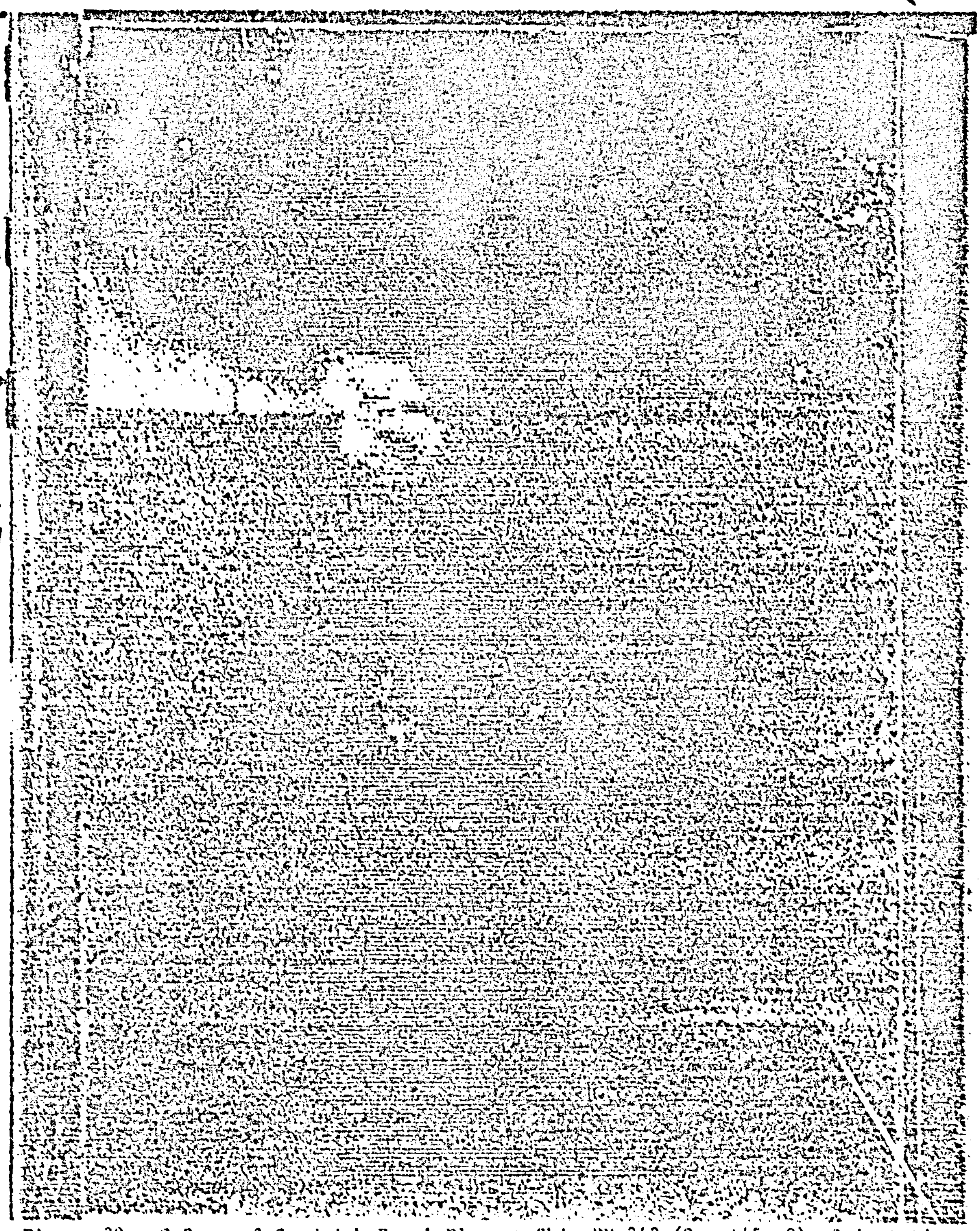


Figure 39. C-Scan of Sandwich Panel Element Skin EX 242 (0_2, ±45, $0)_t$ Orientation

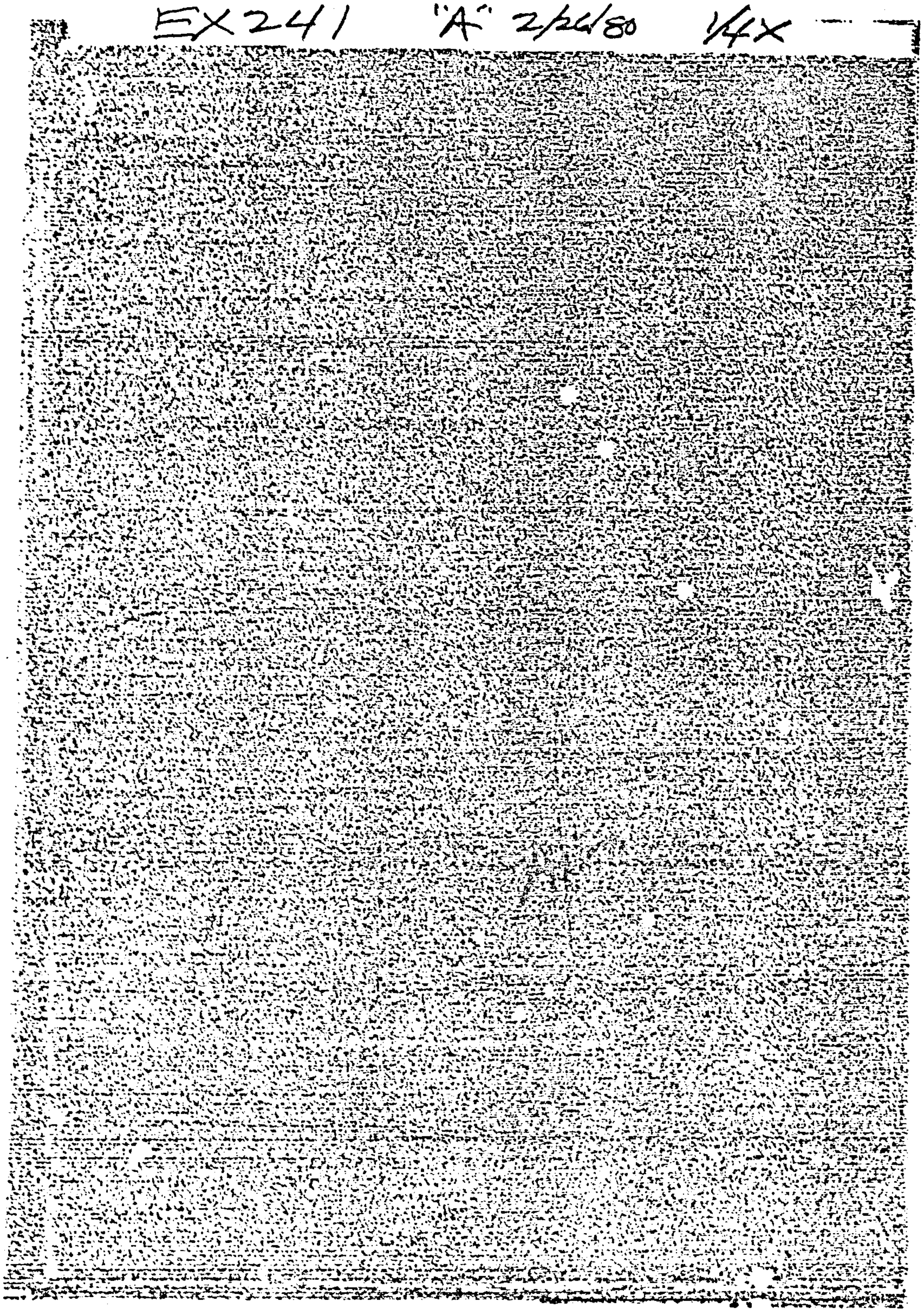


Figure 40. C-Scan of Sandwich Panel Element Skin EX 241 (0_2, ± 45, $0)_t$ Orientation

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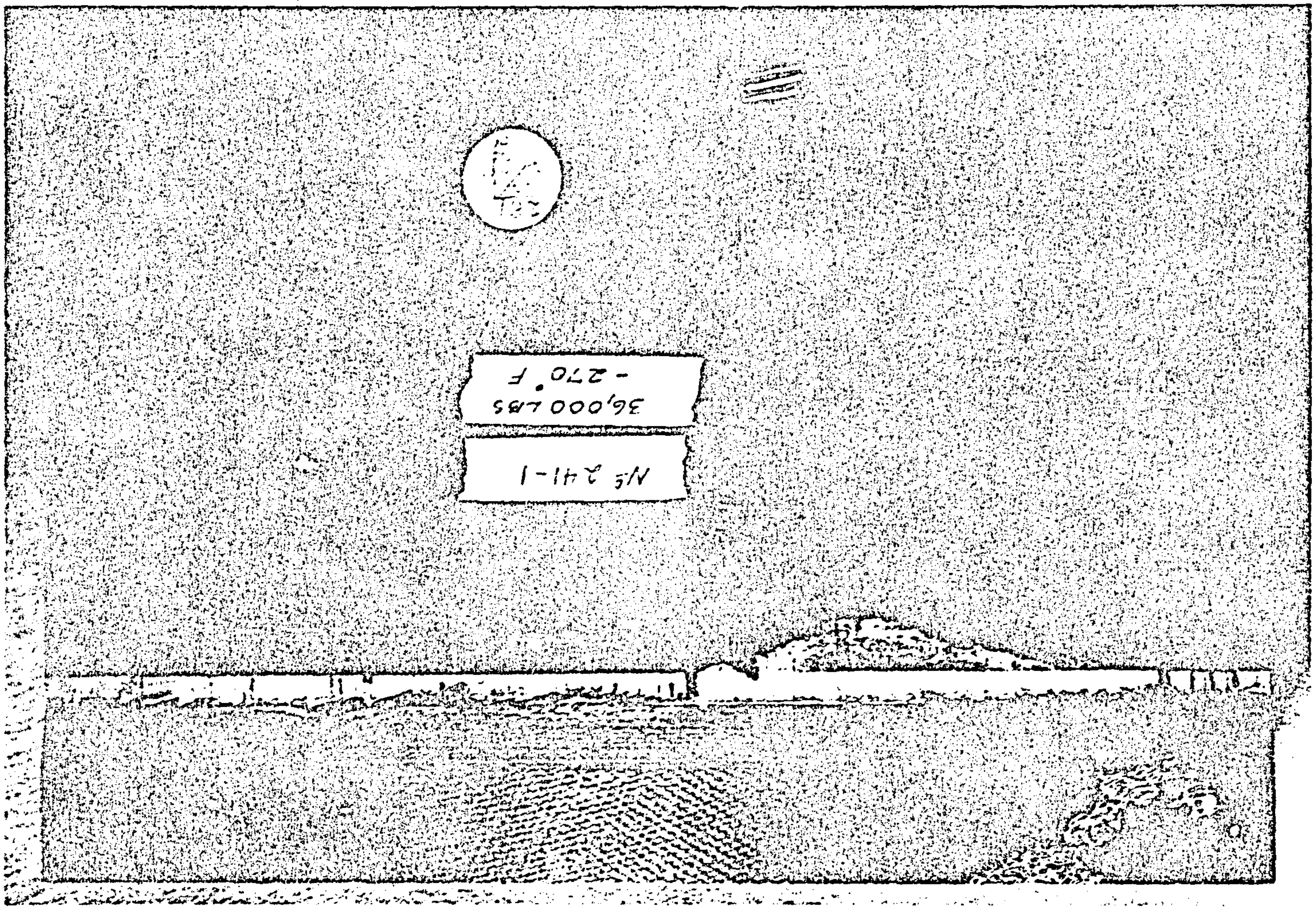

SIDE 1 FAILING LOAD: 160 KN (36,000 LB)

Figure 41. Failure Modes of Sandwich Element EX 241-1, Postcured Condition, Tested at - 132 C (-220 F)

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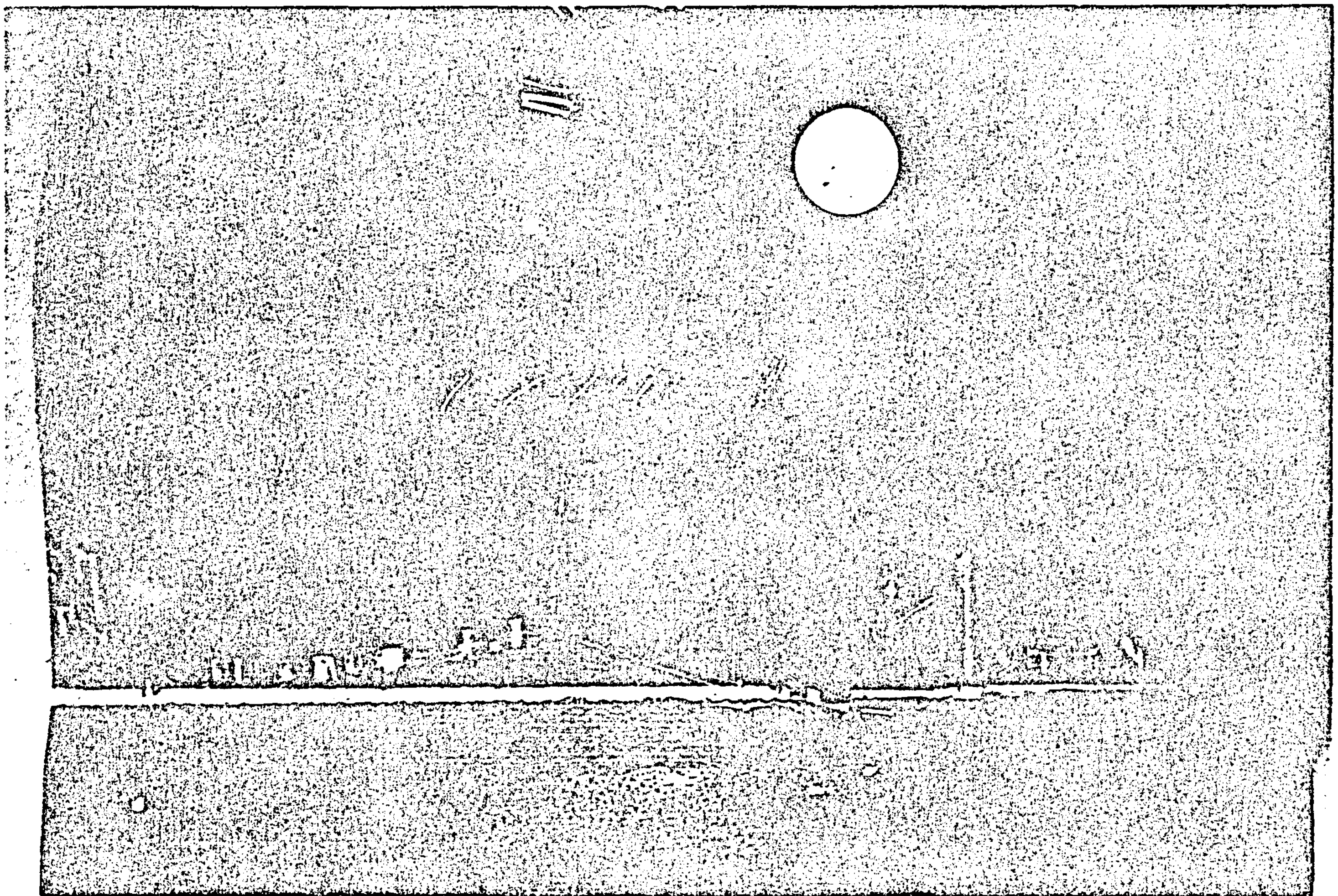

SIDE 2

FAILING LOAD: 160 KN (36,000 LB)

Figure 41A. Failure Modes of Sandwich Element EX 241-1, Postcured Condition, Tested at -132 C (-220 F)

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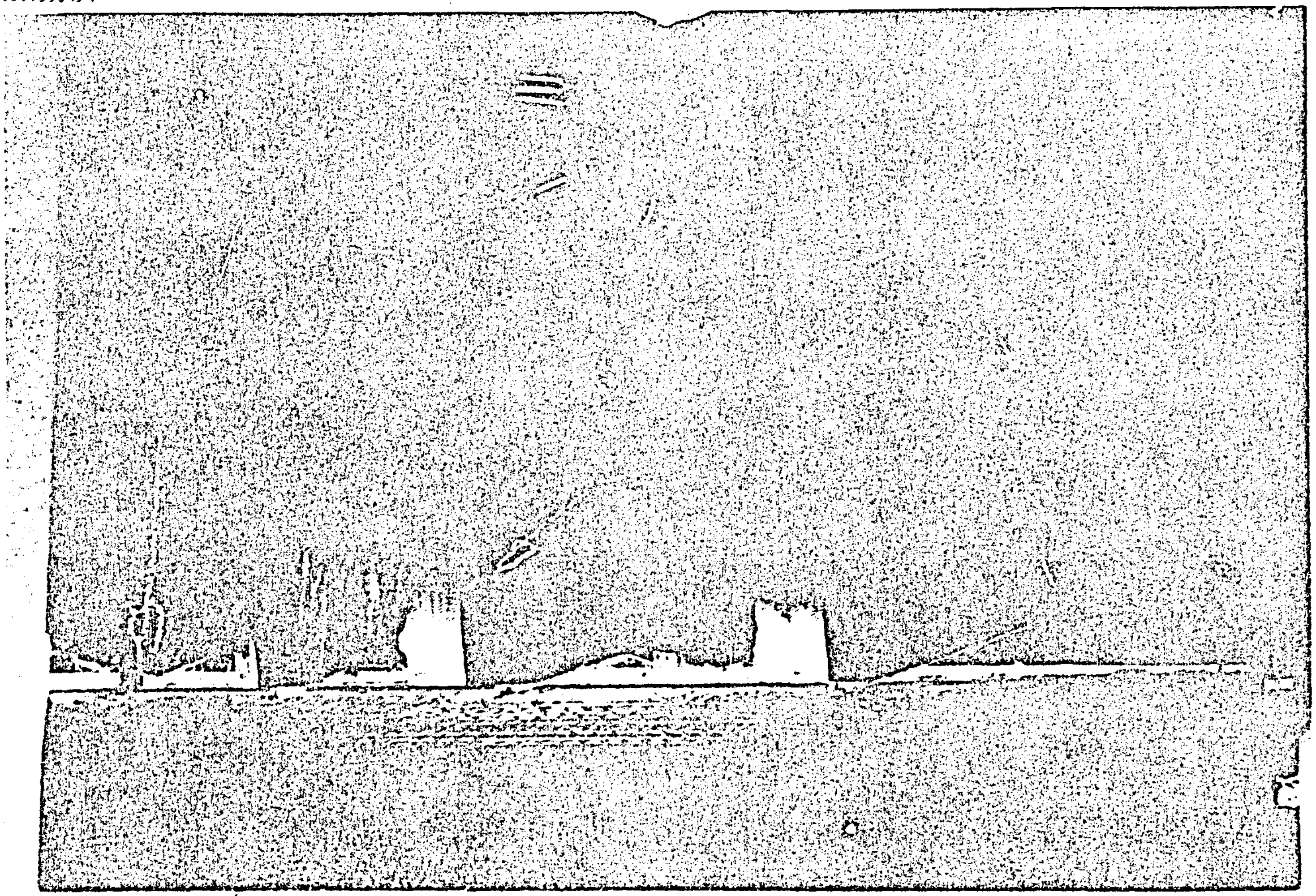
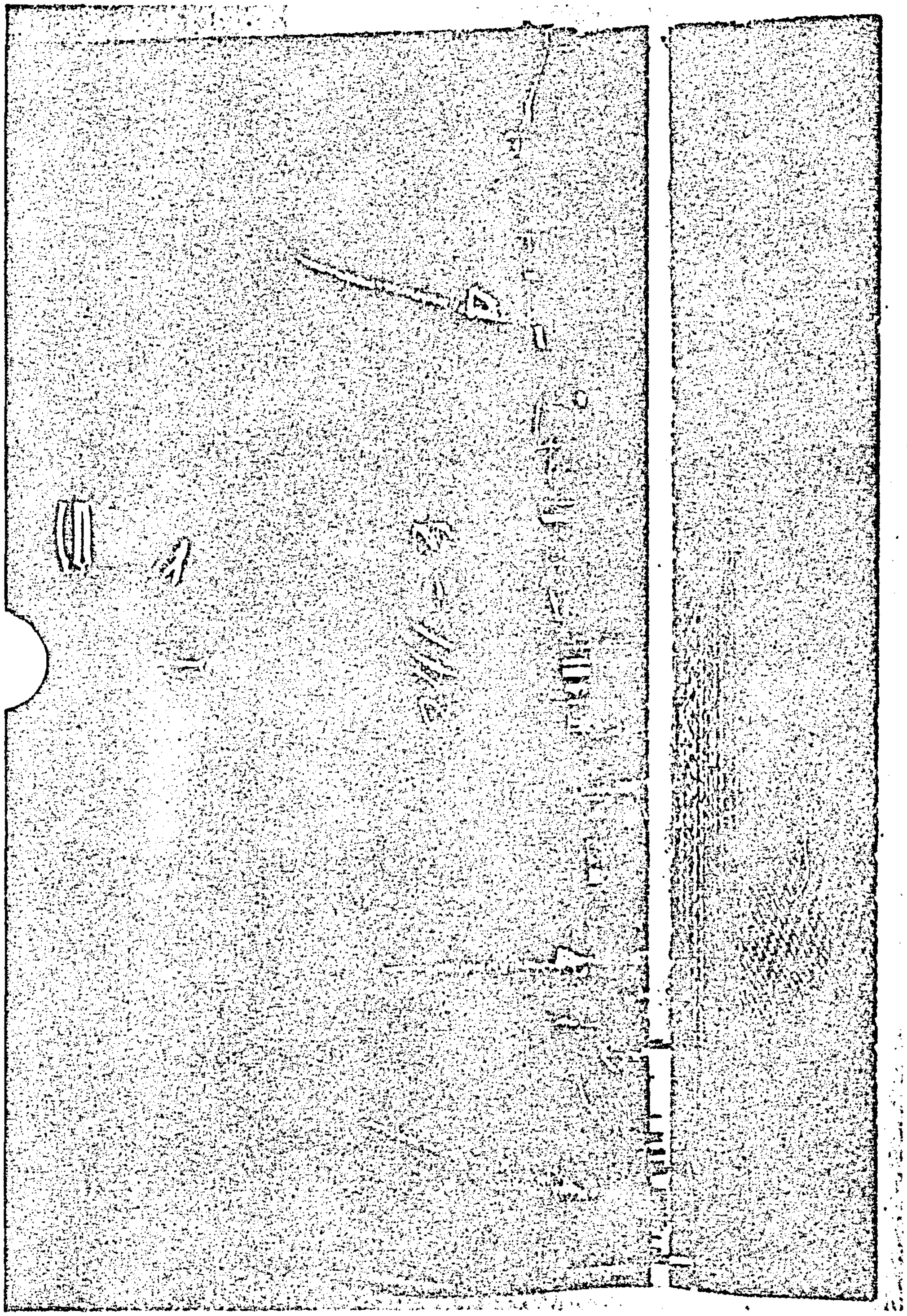


SIDE 1 FAILING LOAD: 156 KN (35,100 LB)

Figure 42. Failure Modes of Sandwich Element EX 241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)



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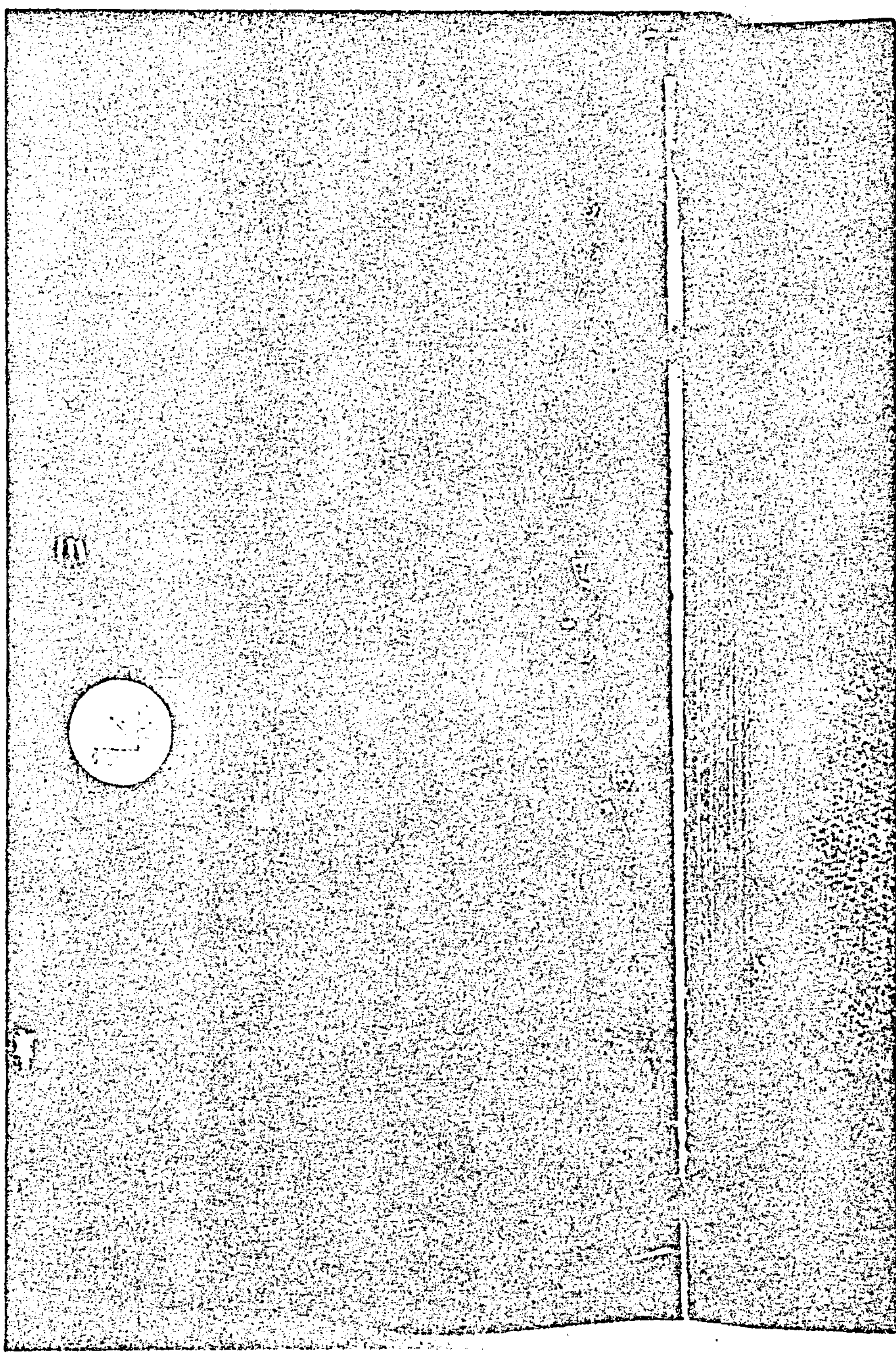
SIDE 2

FAILING LOAD: 156 KN (35,100 LB)

Figure 42A. Failure Modes of Sandwich Element EX 241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)



SIDE 1

FAILING LOAD: 136 KN (30,650 LB)

Figure 43. Failure Modes of Sandwich Element EX 241-2A, Aged 125 Hours at 316 C (600 F), Tested at RT

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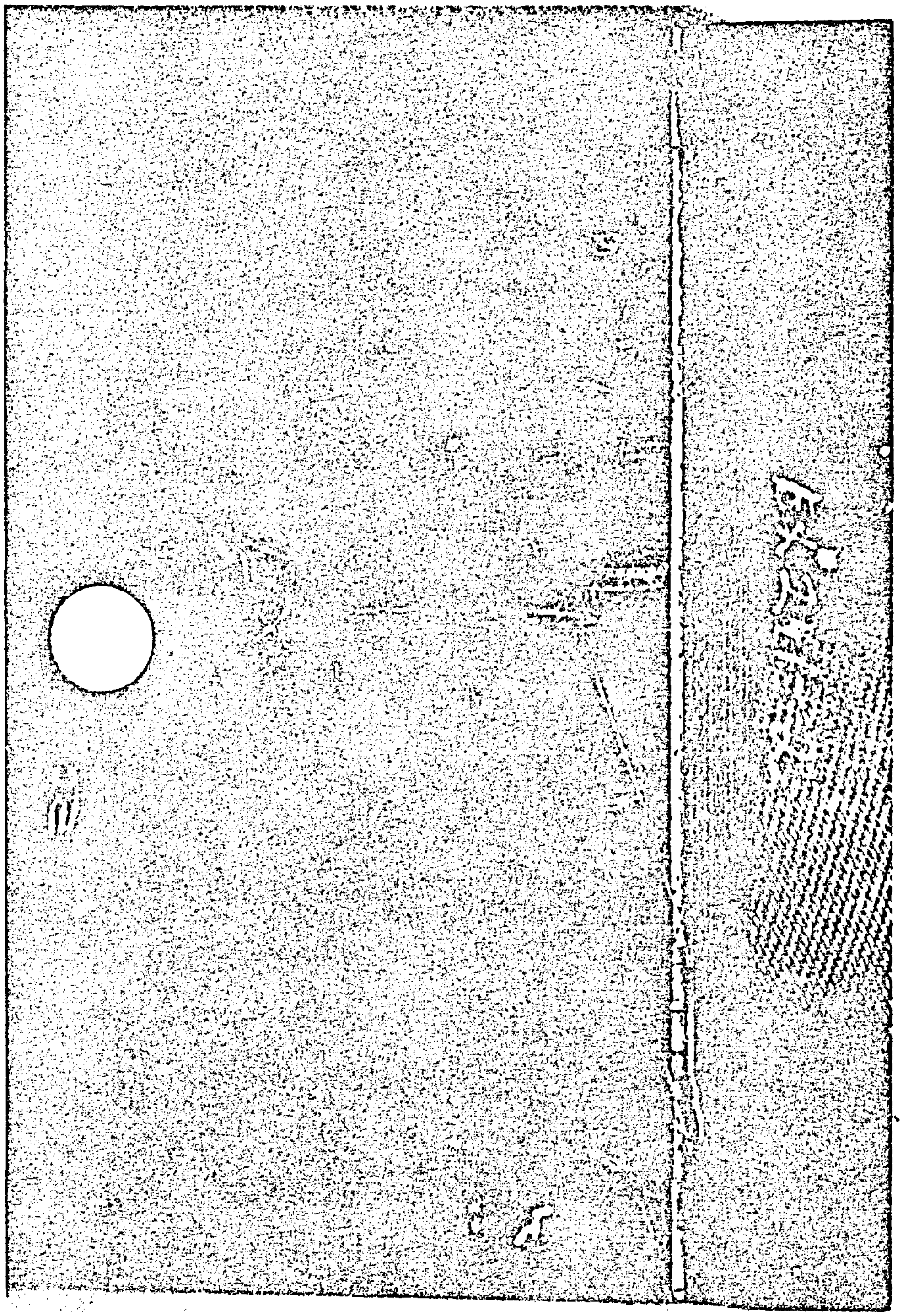

SIDE 2

FAILING LOAD: 136 KN (30,650 LB)

Figure 43A. Failure Modes of Sandwich Element EX 241-2A, Aged 125 Hours at 316 C (600 F), Tested at RT

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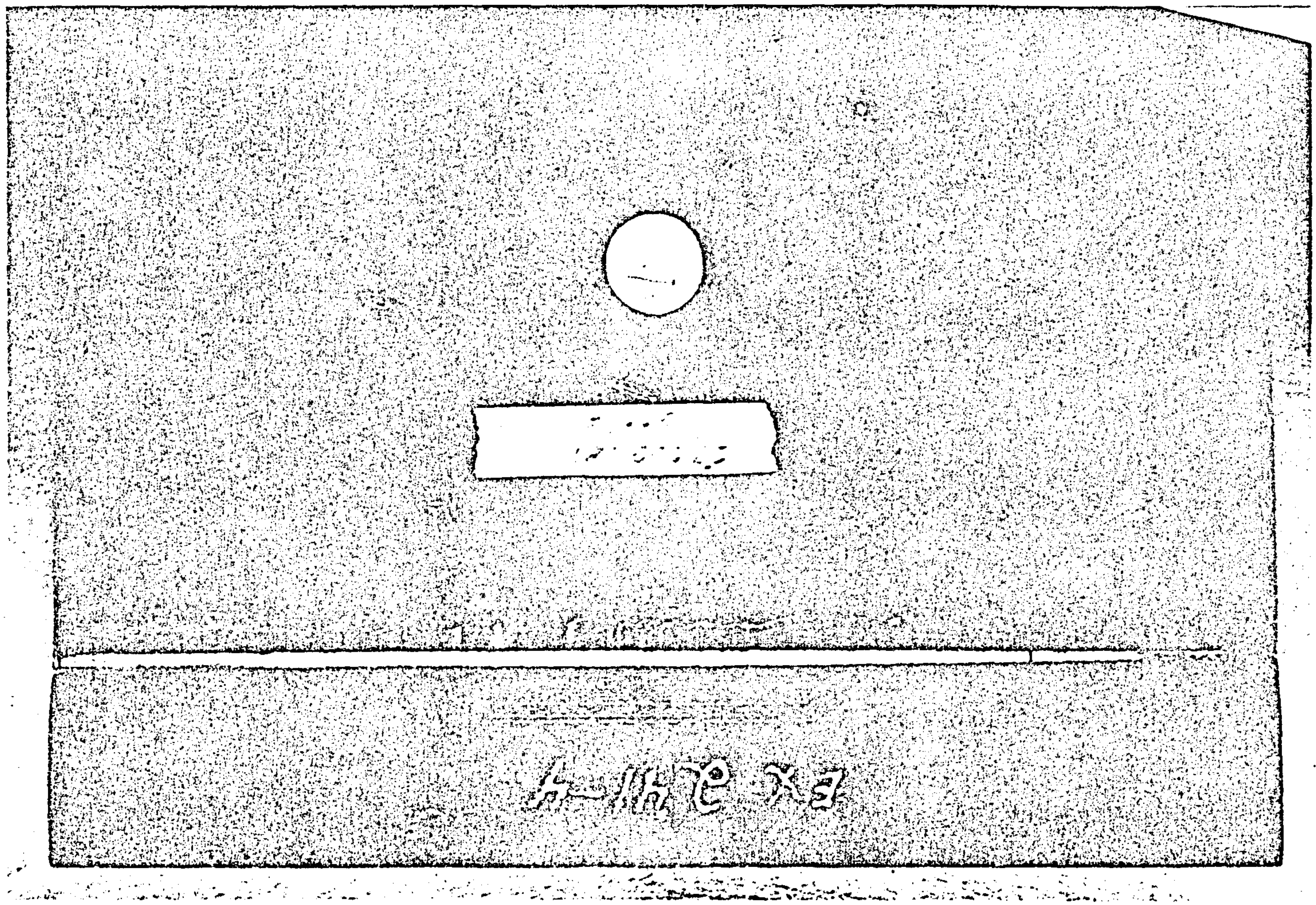
A800808 G-10

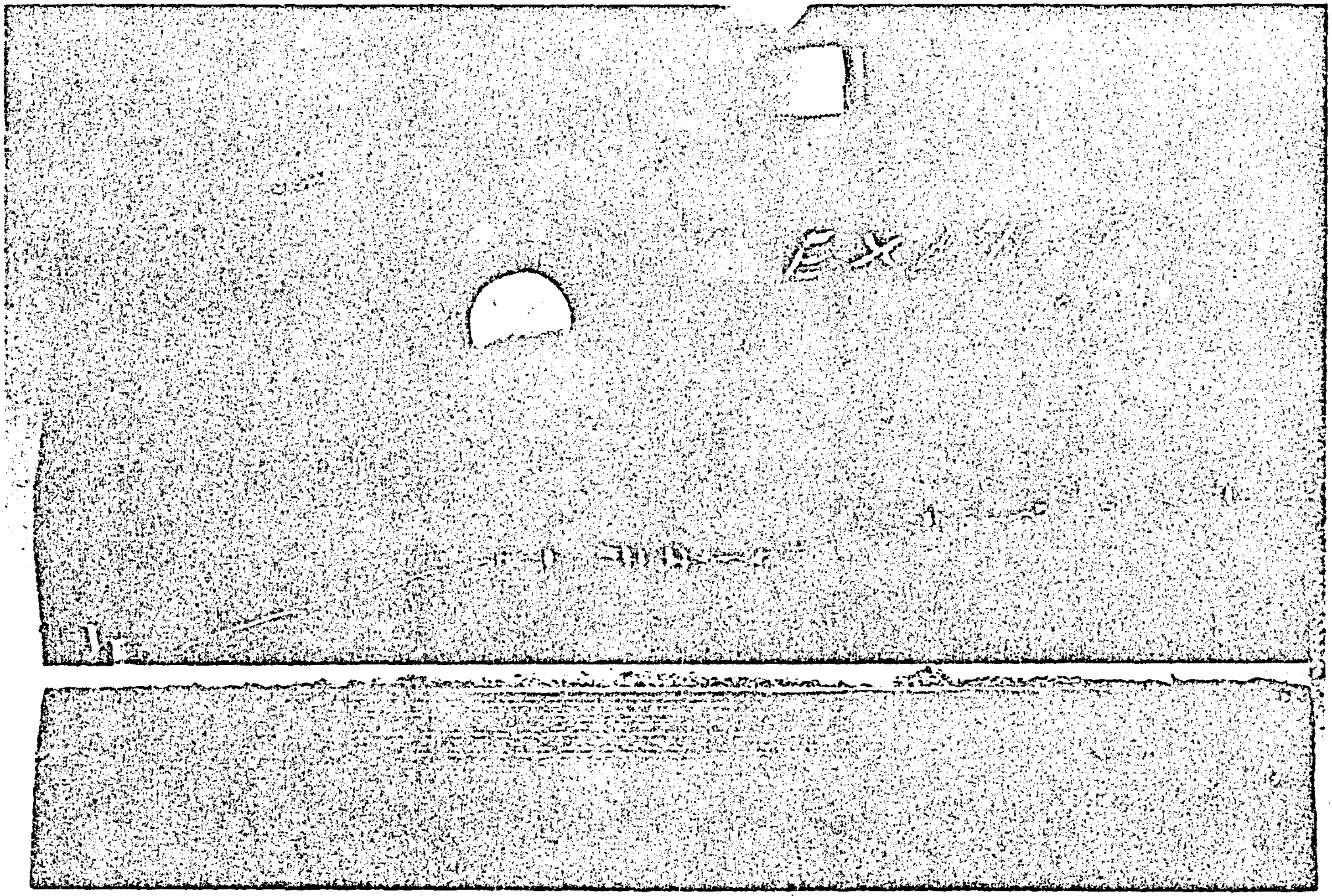


SIDE 1

FAILING LOAD: 120 KN (27,000 LB)

Figure 44. Failure Modes of Sandwich Element EX 241-4A, Aged 125 Hours at 316 C (600 F) Tested at 316 C (600 F)

A800808 G-9



SIDE 2

FAILING LOAD: 120 KN (27,000 LB)

Figure 44A. Failure Modes of Sandwich Element EX 241-4A, Aged 125 Hours at 316 C (600 F) Tested at 316 C (600 F)

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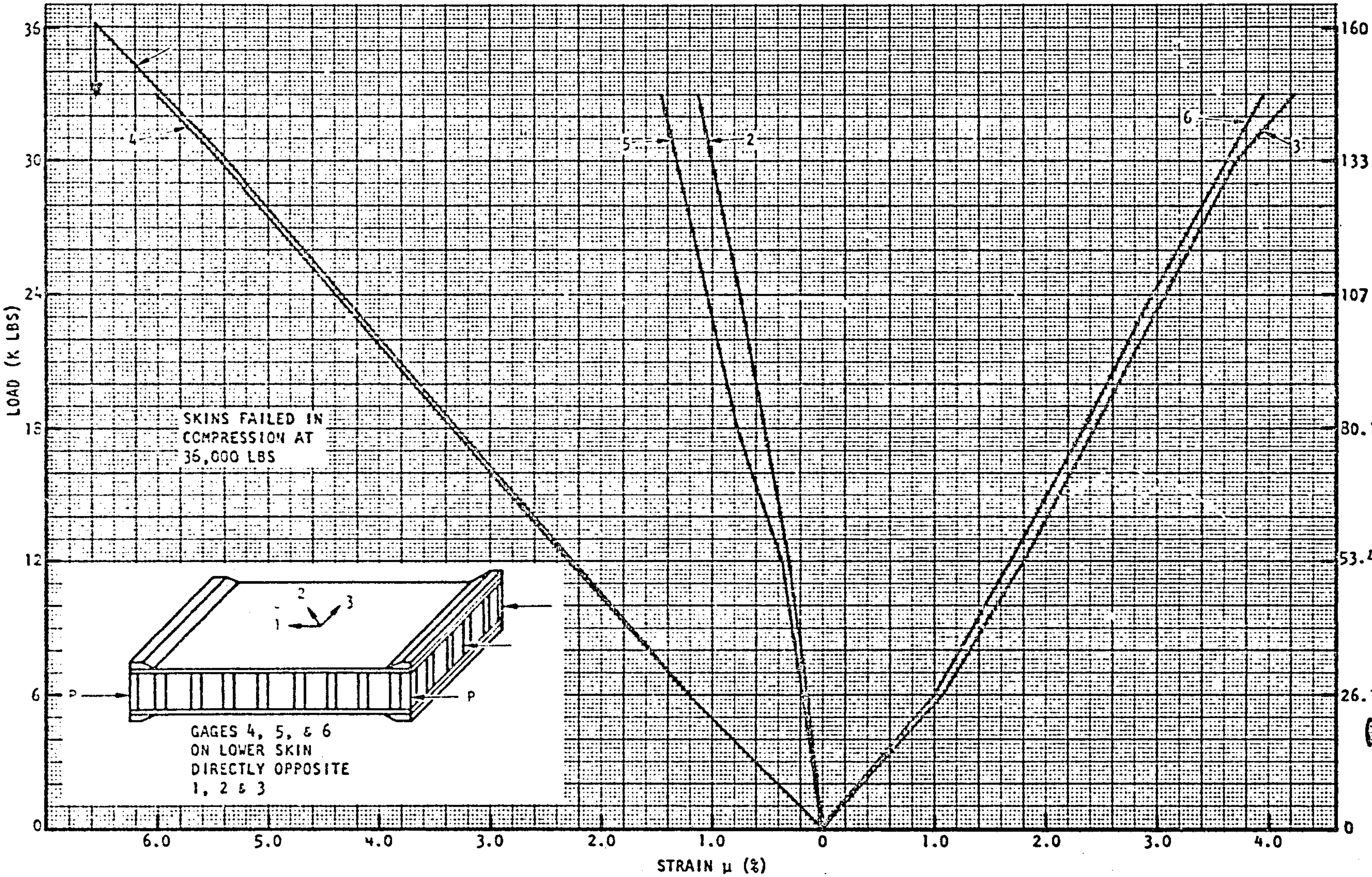

Figure 45. Load/Strain Characteristics of Sandwich Element EX241-1, Postcured Condition, Tested at -132 C (-270 F)

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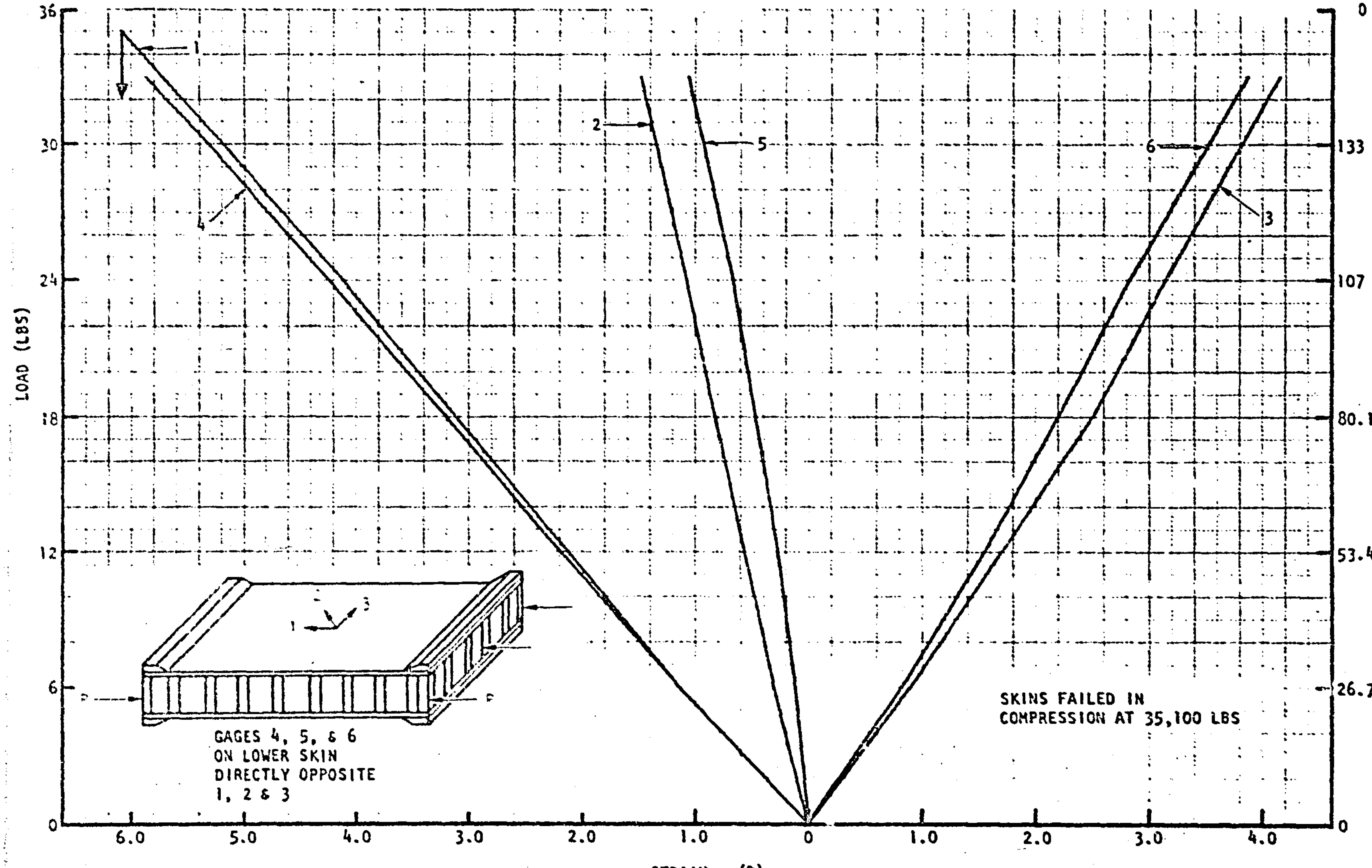

Figure 46. Load/Strain Characteristics of Sandwich Element EX241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)

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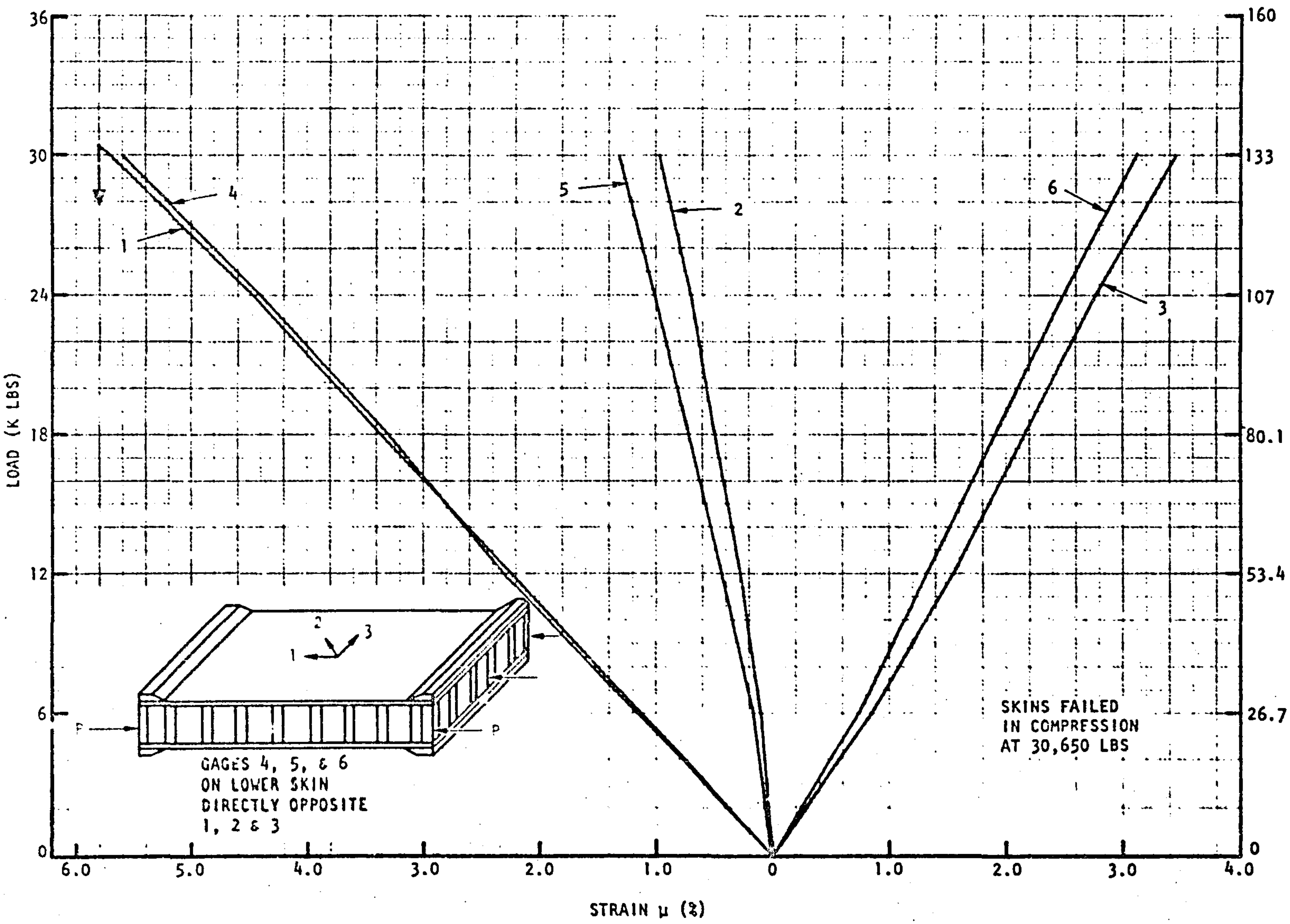

Figure 47. Load/Strain Characteristics of Sandwich Element EX241-2A Aged 125 Hours at 316 C (600 F) Tested at R.T.

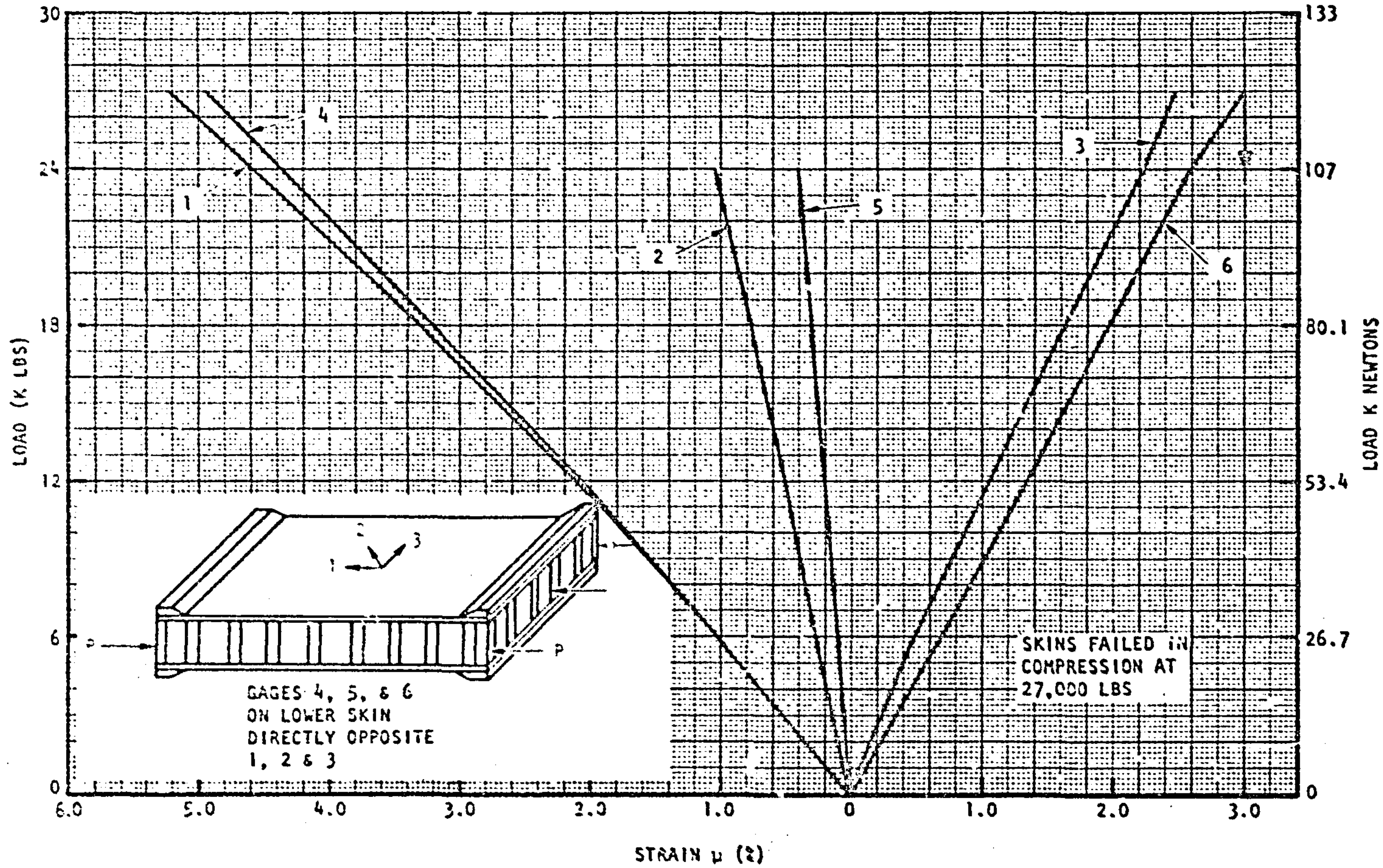

Figure 48. Load/Strain Characteristics of Sandwich Element EX241-4A Aged 125 Hours at 316 C (600 F), Tested at 316 C (600 F)

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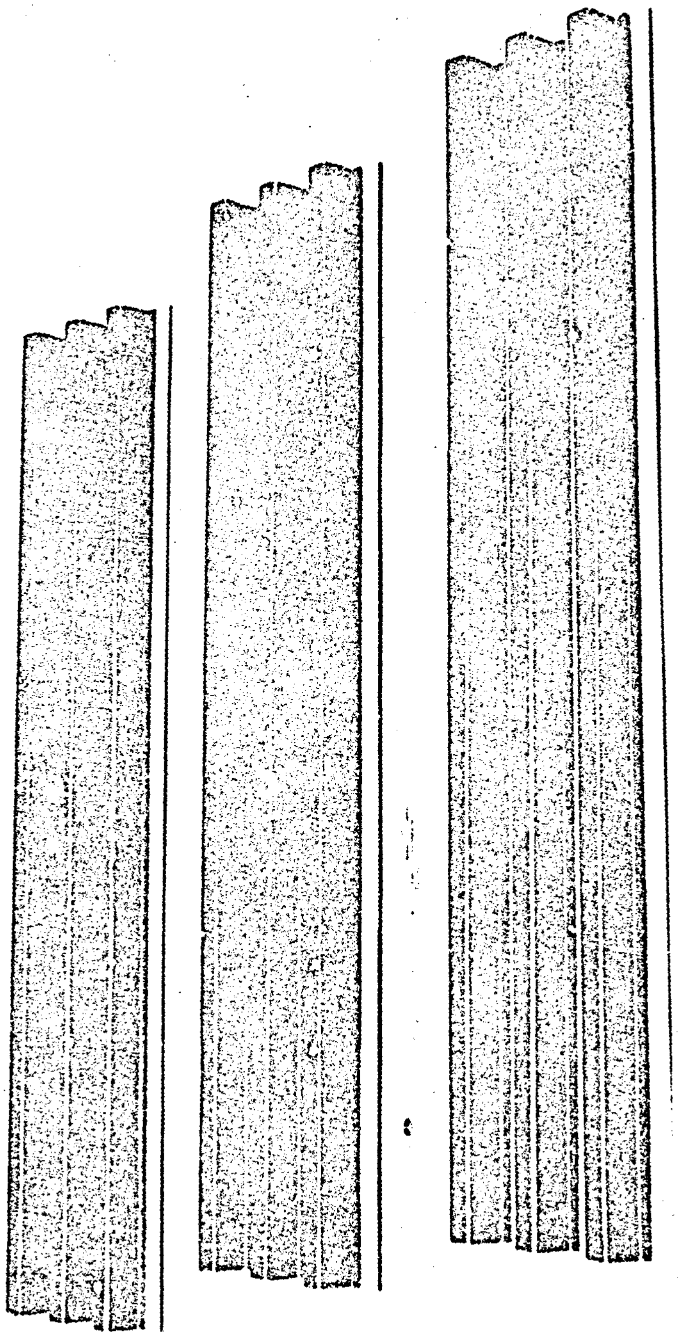

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Figure 49. Three - 122 X 26.0 Cm Hat Stringer/Skin Panels Delivered to NASA-LaRC

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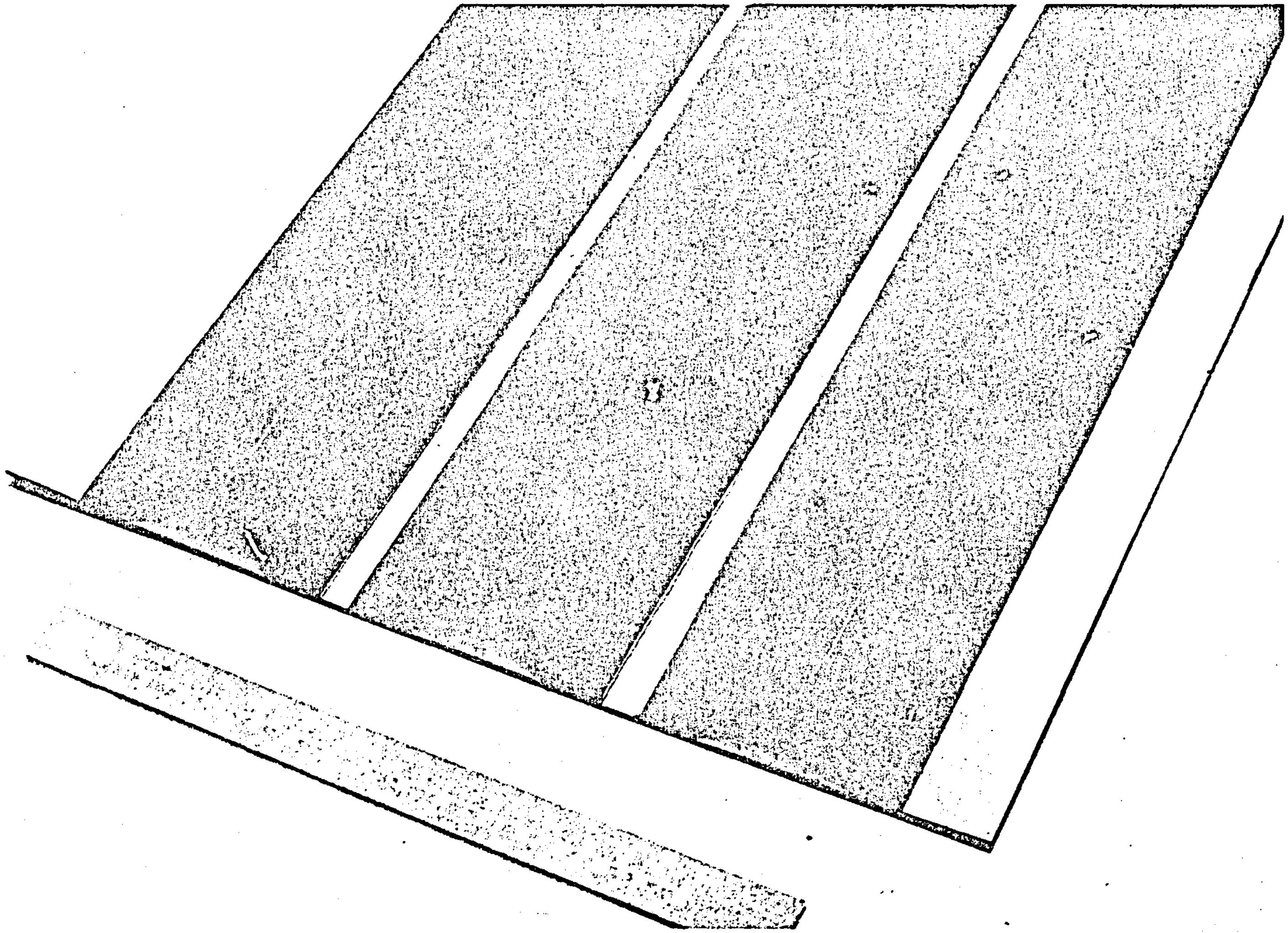

Figure 50. End Views of Hat Stringer/Skin Panels Delivered to NASA-LaRC

A800505F-17C

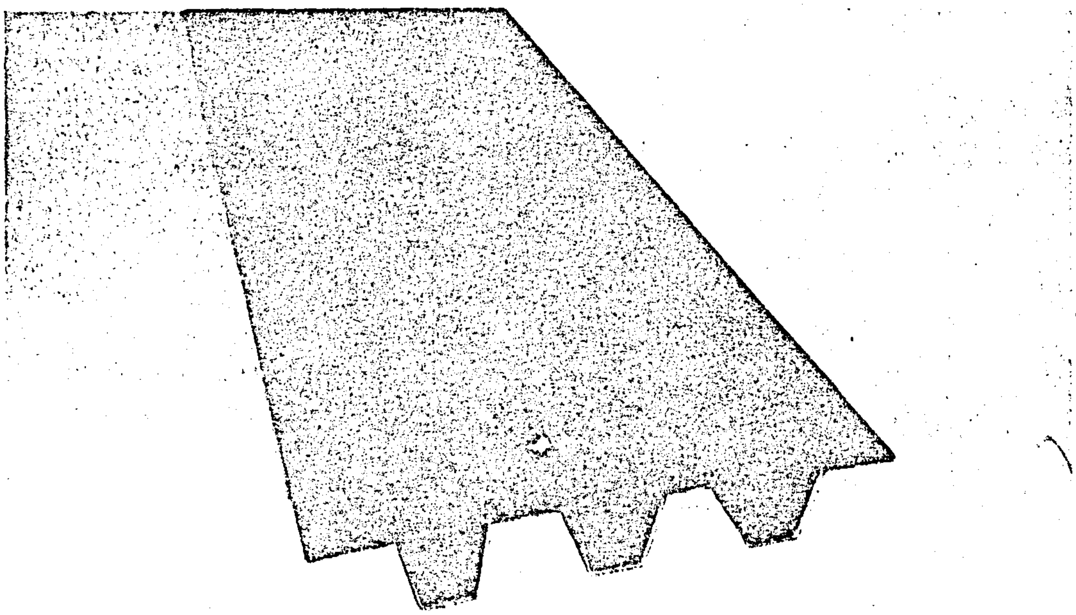

Figure 50A. End Views of Hat Stringer/Skin Panels Delivered to NASA-LaRC

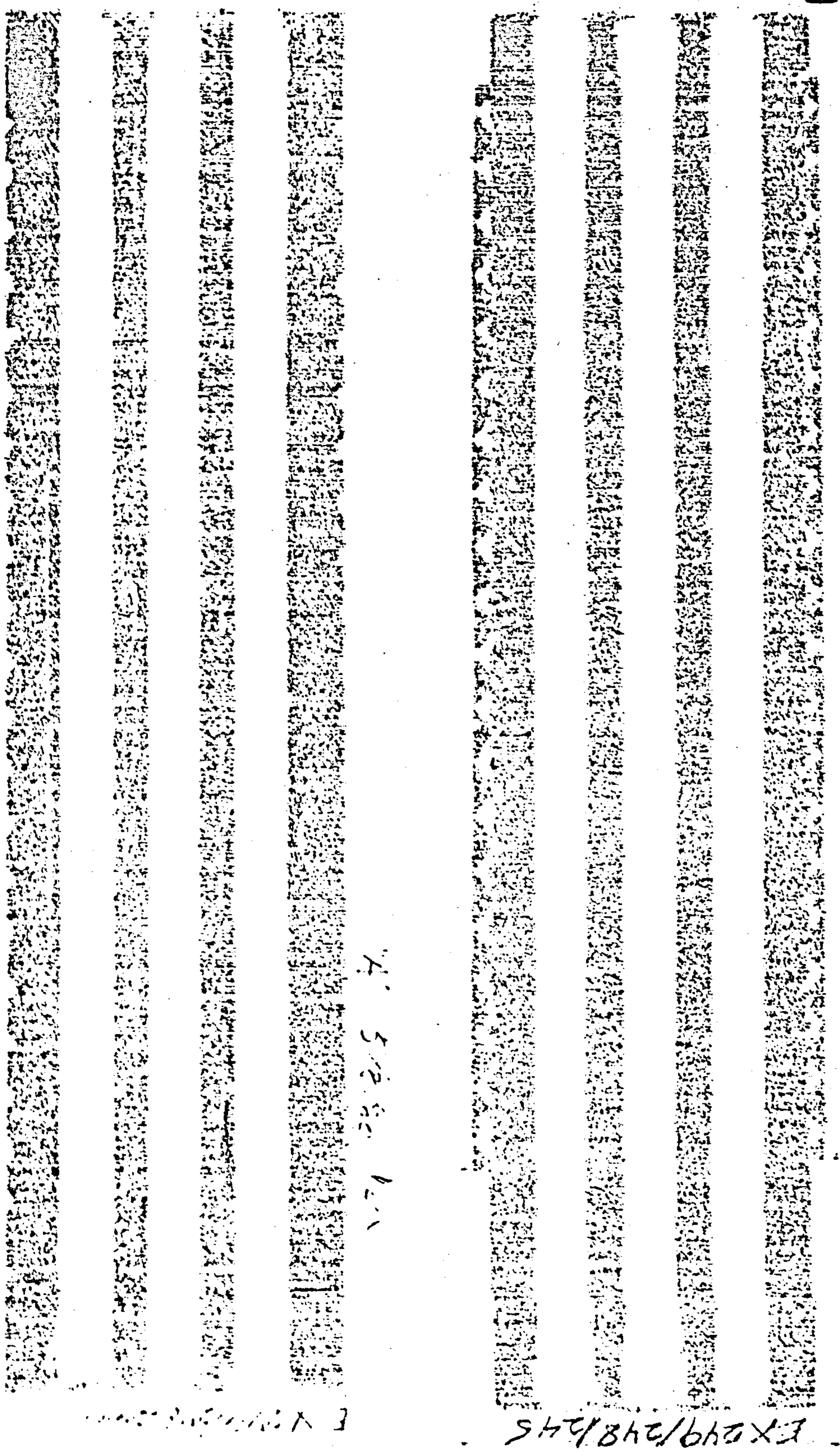

Figure 51. C-Scans of Stringer to Skin Bond Joints, Panels EX249/248/245 and EX279/278/277

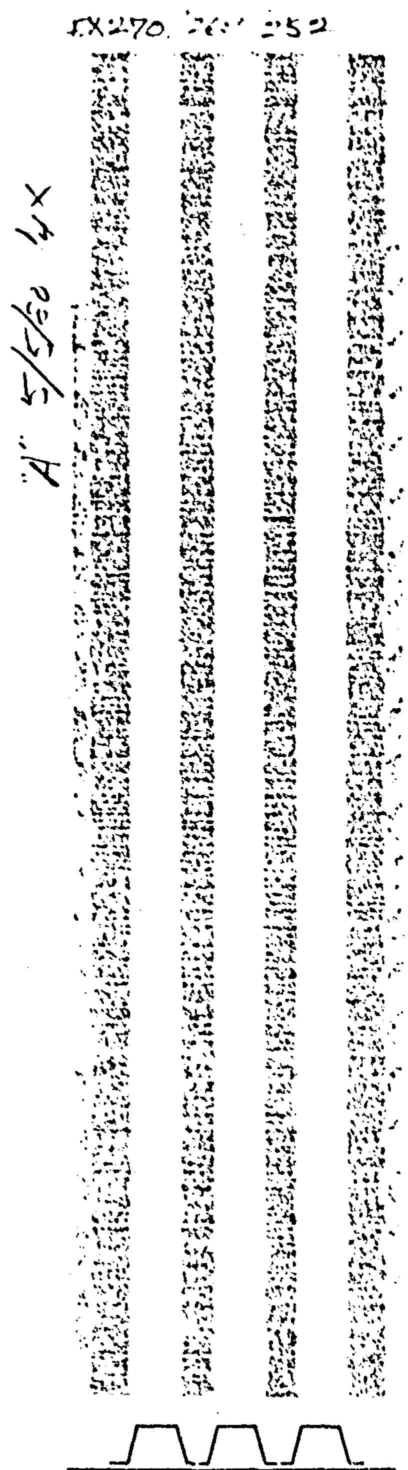

Figure 52. C-Scan of Stringer to Skin Bond Joints, Panel EX270/278/277

Table 1. Imidizing and Cure Cycle Process Improvement Study Observations—Celion 3000/LARC 160 Composites (1)

Imidizing Cycle (2) Observations				Cure Cycle Observations									
Imidize Temp C (F)	Imidize Time (min)	Vol (%)	Resin Solids (%)	Resin Flow Characteristics in Cure	ρ g/cc	Resin Cont (%)	Fiber Vol (%)	Void Vol (%)	Panel Thickness mm	(mils)	Barcol Hardness (Cured)	C-Scan Ultrasound Transmission (%)	Remarks
163 (325)	60	1.64	34.7	high	1.530	26.3	64.4	4.61	1.34-1.63	53-64	72-75	40	Corrugated depressions top and bottom laminate surfaces parallel to fibers. Fiber wash and resin flash excessive on panel sides
	90	1.52	33.0	high	1.531	25.8	64.91	4.20	1.27-1.91	20-25	72-75	40	
	120	1.48	35.0	high	1.545	26.4	64.98	3.64	1.30-1.55	51-61	72-76	40	
	150	1.68	34.0	high	1.55[illegible]	26.8	64.8	3.20	1.14-1.45	45-57	72-75	60	Surface depressions have decreased, fiber wash and resin flash somewhat decreased
	180	1.43	32.6	high	1.563	27.4	64.9	2.21	1.14-1.52	45-60	68-73	50	
177 (350)	60	1.56	35.4	high	1.536	25.7	62.4	4.59	1.22-1.65	48-65	72-74	50	Corrugated depressions top and bottom surfaces parallel to fibers. Fiber wash and resin flash excessive on panel sides
	90	1.15	35.2	high	1.521	28.2	65.2	4.42	1.22-1.52	48-60	72-75	50	
	120	1.45	34.4	high	1.540	27.1	64.2	3.74	1.35-1.50	53-59	73-76	75	Laminate surfaces smooth; same fiber wash and resin flashing on sides
	150	1.06	34.6	high	1.565	28.0	64.4	1.91	1.22-1.55	48-61	74-78	95	
	180	1.14	34.2	high	1.561	28.5	63.8	2.01	1.27-1.65	50-65	73-77	95	
191 (375)	30	0.73	34.9	l[illegible]	1.576	30.0	63.0	0.56	1.50-1.57	59-62	72-77	95	Very minor top and bottom surface depressions and edge fiber washing
	60	1.22	34.6	med	1.575	31.5	61.6	0.19	1.52-1.63	60-64	74-78	95 (2 splices)	Top and bottom surfaces very smooth and uniform, minor fiber washing
	90	1.24	35.0	med	1.570	33.3	59.8	-0.04	1.52-1.60	60-63	74-77	95 (1 splice)	
	120	1.17	33.8	med	1.574	33.6	59.7	-0.04	1.50-1.60	59-63	75-79	95 (1 splice)	
	150	0.92	34.9	med	1.565	35.0	58.1	-0.25	1.57-1.65	62-65	72-77	100	
199 (390)	30	1.21	35.0	med	1.577	32.3	61.0	-0.19	1.50-1.60	60-63	75-77	100	Top and bottom surfaces very smooth and uniform. minor fiber washing
	60	0.87	35.1	med	1.563	34.9	58.1	-0.10	1.60-1.65	63-65	74-76	100	
	90	1.10	33.3	low	1.579	33.7	59.8	-0.76	1.57-1.63	62-64	73-75	100	
	120	0.71	34.2	low	1.560	34.6	58.3	0.18	1.63-1.65	64-65	73-76	100	
	150	0.66	36.7	low	1.562	34.8	58.2	-0.015	1.60-1.68	63-66	70-76	100	
218 (425)	30	1.29	34.7	low	1.568	33.1	59.9	0.13	1.55-1.63	61-64	74-77	100	Top and bottom surfaces very smooth and uniform, minor fiber washing
	60	1.35	33.2	low	1.560	33.8	59.0	0.43	1.55-1.63	61-64	72-76	100	
	90	1.37	35.2	low	1.565	34.1	58.9	-0.011	1.57-1.60	62-63	74-76	100	
	120	1.15	34.0	low	1.550	35.1	56.8	-0.042	1.60-1.68	63-66	73-76	100	Top and bottom surfaces smooth but appear resin rich due to lack of flow, minor fiber wash and resin flash
	150	1.16	35.2	low	1.553	35.5	57.2	0.35	1.60-1.65	63-65	72-77	95	

(1) Celion 3000 fiber (epoxy resin sized) employed by U.S. Polymeric Inc. in making 30.4 cm (12.0 inches) wide, nominal 67 ±3 grams/m² areal fiber weight prepreg. Prepreg—LARC 160 resin solids: 38 ±3%; volatiles: 12 ±3%.

(2) One ply 120 fiberglass top and 1 ply bottom surfaces used in imidizing and cure cycles to absorb excess resin to target 60 ±2 composite fiber volume.

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Table 2. Test Matrix--LARC 160 Imidizing and Cure Cycle Process Improvement Study

Imidizing Temperature C (F)	Imidizing Time (Mins)				
163 (325 F)	60	90	120	150	180
177 (350 F)	60	90	120	150	180
191 (375 F)	30	60	90	120	150
199 (390 F)	30	60	90	120	150
218 (425 F)	30	60	90	120	150

Total: 25-6 X 6 - 26 ply 0° onidirectional laminates comprised of Celion 3000 (epoxy sized/LARC 160, U.S. Polymeric Inc batch 2W4612-2 prepreg.

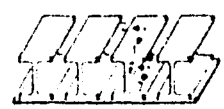
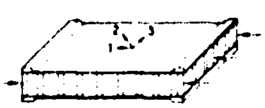
Table 3. Prepreg and Composite Physical Properties, Celion/LARC 160 Standard Imidizing and Cure Process Verification(1)

Prepreg Batch No. and Panel No.	Prepreg Physical Properties: Resin Solids (%)	Vols (%)	Fiber Area Wt (g/m²)	Calculated T/Ply(2): mm	(mils)	No./Type of Fiberglass Bleeder Plies: Top	Bottom	Composite Physical Properties: Density gram/cc	Resin Wt Content (%)	Fiber Volume (%)	Void Volume (%)	No. Plies	Thickness/Ply: mm	(mils)	Total Thickness: mm	(mils)	C-Scan Ultrasound Transmission (%)	Remarks
CSP 4612-2	39.3	11.8	65.5	0.061	(2.44)	1/120	1/120	1.555	35.9	57.0	0.09	32	0.063-0.068	2.53-2.69	2.06-2.18	81-86	100	Standard LARC 160 resin, Celion 3000 fiber is epoxy resin sized
FIB. C0161	33.4	9.7	163.4	0.155	(6.1)	0	0	1.556	33.8	58.9	0.68	16	0.109-0.116	4.31-4.56	1.76-1.86	69-73	100	Standard LARC 160 resin, XR15082 resin sizing on Celion 6000 fiber
FIB. C0261	35.4	9.9	[illegible]	0.143	(5.63)	0	1/120	1.560	36.1	57.0	-0.28	16	0.108-0.113	4.25-4.43	1.73-1.80	68-71	100	Standard LARC 160 resin, XR15082 resin sizing on Celion 6000 fiber
HEX 22945	42.2	14.3	131	0.127	(5.0)	1/120	1/120	1.544	34.2	58.8	0.04	16	0.124-0.132	4.88-5.19	1.98-2.11	78-83	100	Resin variable: +5% AP22
HEX 22946	40.4	15.2	127	0.122	(4.8)	1/120	1/120	1.537	39.3	53.3	0.22	16	0.122-0.128	4.81-5.06	1.96-2.06	77-81	100	Resin variable: -5% AP22
HEX 22949	40.0	14.1	122	0.117	(4.6)	2/120	2/120	1.552	36.2	56.6	0.21	16	0.119-0.124	4.69-4.88	1.90-1.98	75-78	100	Resin variable: NA +5%, BTDA-std
HEX 22950	42.0	14.0	122	0.117	(4.6)	1/120	1/120	1.556	38.1	55.0	-0.66	16	0.124-0.127	4.89-5.00	1.98-2.03	78-80	100	Resin variable: NA -5%, BTDA-std
HEX 22951	43.0	14.3	116	0.112	(4.4)	1/120	2/120	1.543	38.2	54.5	0.19	16	0.121-0.125	4.75-4.93	1.93-2.01	76-79	100	Resin variable: NA std, BTDA +5%
HEX 22952	42.1	14.8	120	0.112	(4.4)	2/120	2/120	1.559	33.3	58.9	0.65	16	0.114-0.127	4.50-5.00	1.83-2.03	72-80	100	Resin variable: NA std, BTDA -5%
HEX 22954	40.5	12.9	120	0.114	(4.5)	2/120	1/120	1.560	35.6	57.5	-0.10	16	0.124-0.133	4.88-5.25	1.98-2.13	78-84	100	Resin variable: cook time 2 hours at 60°C
HEX 22955	40.0	14.8	121	0.117	(4.6)	2/120	2/120	1.555	36.1	56.8	0.04	16	0.121-0.127	4.75-5.00	1.93-2.03	76-80	100	Resin variable: reflux time 6 hours

(1) Standard imidizing and autoclave cure processes described in the 7th Quarterly Report were employed in laminate fabrication. Laminate size: 30.4 x 30.4 cm (12 x 12 inches); orientation: (0, ±45, 90)s.

(2) Calculated "T"/ply based on target 60 percent fiber volume.

Table 4. Results of Compression Tests on "Hat" and "I" Stiffened Skin and Sandwich Panel Structural Elements(1)

Element Configuration	Condition(2)	Element No.	Test Temperature C	Test Temperature (F)	Ultimate Load KN	Ultimate Load (LBS)	Remarks
 STRAIN GAGES 1 AND 2 INSTALLED ON LOWER SKIN DIRECTLY OPPOSITE 3 AND 4	1	EX109/EX110A	24	(75)	120.8	(27,150)	Achieved design ultimate, compressive failure of hat caps with transfer thru webs occurred during strain gage readout at 27,150 lbs. Skin and bond failures were secondary.
		EX109/EX110B	24	(75)	120.8	(27,150)	Achieved design ultimate - no failure. Specimen was then fatigue tested 5% to 67% of design ultimate, compression/compression load to 265,000 cycles - no failure.
		EX109/EX110B	-132	(-270)	116.8	(26,250)	Skin compression failure - load dropped to 19,500 lbs. Retest of EX109/EX110B specimen tested at RT. Failed at 97% of design ultimate.
		EX195-1 PC	316	(600)	87.84	(19,750)	Skin compression failure, bottom 2 corners followed by buckling through bottom-center. Minor bond failure, center stringer under skin buckle. 73% of RT design ultimate.
	2	EX195-2A	24	(75)	120.8	(27,150)	Achieved design ultimate - no failure.
		EX195-4A	-132	(-270)	120.8	(27,150)	Achieved design ultimate - no failure.
		EX195-3A	316	(600)	124.3	(27,950)	Skin compression failure inboard of 1 bottom corner followed by diagonal skin buckling toward center of panel. Minor debond under center stringer. Failed at 103% of RT design ultimate.
 STRAIN GAGES 3 AND 4 INSTALLED ON LOWER SKIN OPPOSITE 1 AND 2	1	EX111/EX113	24	(75)	125.4	(28,187)	Achieved design ultimate - no failure.
		EX111/EX113	-132	(-270)	125.4	(28,187)	Achieved design ultimate - no failure. Retest of EX111/EX113 tested at RT.
		EX111/EX113	24	(75)	125.4	(28,200)	Achieved design ultimate - no failure. Retest of EX111/EX113 tested at RT and -270 F.
		EX194-1 PC	316	(600)	125.4	(28,187)	Achieved design ultimate - minor skin compression failure in 1 corner. Did not cause drop in load. No debonds.
	2	EX194-2A	24	(75)	125.4	(28,187)	Achieved design ultimate - no failure.
		EX194-4A	-132	(-270)	125.7	(28,250)	Achieved design ultimate. Compression failure of skin starting at 1 upper corner extends inboard 1 inch. Two stringer caps and webs also failed in compression with caps splitting axially. No debonds.
		EX194-3A	316	(600)	125.4	(28,187)	Achieved design ultimate - no failure.
 STRAIN GAGES 4, 5, AND 6 INSTALLED ON LOWER SKIN OPPOSITE 1, 2, AND 3	1	EX150-1	24	(75)	125.7	(28,[illegible])	Achieved required 0.53 MN/m (3000 lbs/inch) compression load. Skin compression failures occurred along edges of bottom doublers. No debonds.
		EX241-1PC(3)	-132	(-270)	160.1	(36,000)	Achieved 133% RT design ultimate. Compression failure both skins next to doubler.
		EX150-2	316	(600)	97.86	(22,000)	Skin compression 1 side only, top corner, 1.12 inch above doubler at edge, extends 1.0 inches inboard. Achieved 81% of RT requirement of 0.53 MN/m (3000 lbs/inch). No debonds.
	2	EX241-2A(3)	24	(75)	136.3	(30,650)	Achieved 113% RT design ultimate. Compression failure both skins next to doubler; also core shear failure after skin failure due to instability.
		EX241-3A(3)	-132	(-270)	156.1	(35,100)	Achieved 130% RT design ultimate. Compression failure both skins 1.0 inch above doubler and next to doubler.
		EX241-4A(3)	316	(600)	120.1	(27,000)	Achieved 100% RT design ultimate. Compression failure 1 skin next to doubler and 1 skin 1.0 inch above doubler.

(1) Specimen designs are given in 2nd and 3rd quarterly reports. Load/Strain curves are presented in Figures 65 through 79

(2) Condition 2: Postcured 4 hours at 316 C (600 F); 2 aged 125 hours at 316 C (600 F)

(3) New data

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